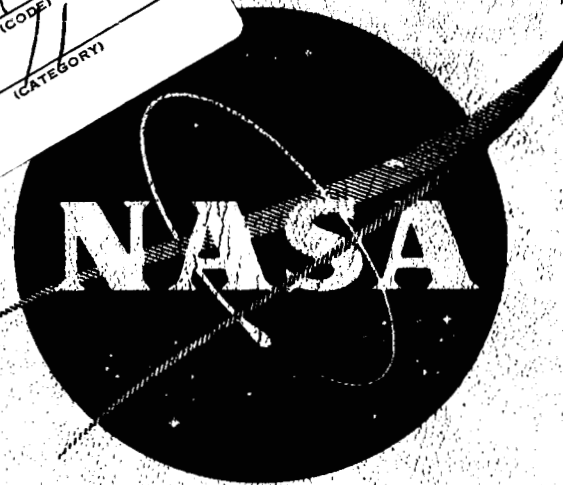NASA-CR-72042

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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

Quarterly Progress Report No. 11
For Quarter Ending April 15, 1966

EDITED BY E.E. HOFFMAN

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SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ⚡ ELECTRIC
CINCINNATI, OHIO 45215

POTASSIUM CORROSION TEST LOOP DEVELOPMENT

QUARTERLY PROGRESS REPORT 11

Covering the Period

January 15, 1966 to April 15, 1966

Edited by

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Lewis Research Center

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Technical Management
NASA - Lewis Research Center
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SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
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I. INTRODUCTION

This report covers the period, from January 15, 1966 to April 15, 1966, of a program to develop a Prototype Corrosion Test Loop for the evaluation of refractory alloys in boiling and condensing potassium environments which simulate project space electric power systems. The prototype test consists of a two-loop Cb-1Zr facility; sodium is heated by direct resistance in the primary loop and is used in a heat exchanger to boil potassium in the secondary corrosion test loop. Heat rejection for condensation in the secondary loop is accomplished by radiation in a high-vacuum environment. The immediate corrosion test design conditions are shown below; it is expected that the temperature could be increased by about 400°F when testing is extended to include refractory alloys stronger than Cb-1Zr.

1. Boiling temperature, 1900°F

2. Superheat temperature, 2000°F

3. Condensing Temperature, 1350°F

4. Subcooling temperature, 800°F

5. Mass flow rate, 20 to 40 lb/hr

6. Vapor velocity, 100 to 150 ft/sec

7. Average heat flux in the potassium boiler - 50,000 to 100,000 BTU/hr ft^2

The development program includes the construction and operation of three Cb-1Zr test loops, each of which are being used in a sequence of component evaluation and endurance testing. Loop I, a natural convection loop, has been operated for 1,000 hours with liquid sodium at a maximum temperature of 2260°F to 2380°F to evaluate the electrical power vacuum feedthroughs, thermocouples, the method of attaching the electrodes, the electrical resistivity characteristics of the heater segment, and the use of thermal and electrical insulation. Loop II, a single-phase sodium, forced-circulation loop to evaluate the primary loop EM pump, a flowmeter, flow control and isolation valves, and pressure transducers, has completed 2,650 hours of scheduled testing. This loop was operated at a maximum temperature of 2065°F and a pump inlet temperature of 1985°F. The Prototype Corrosion Test Loop, a two-loop system, includes a boiler, turbine simulator and condenser in addition to the above components. This facility was used to develop and endurance test (5,000 hours) the components required to achieve stable operation at the corrosion test design conditions. The 5,000-hour test has been completed and the evaluation phase of the program has been initiated.

II. SUMMARY OF PROGRESS

The Prototype Corrosion Loop was returned to the test conditions on January 18, 1966, following the shutdown on January 8. The loop operated in a very stable and uneventful manner during the remainder of the 5,000-hour test. The shutdown on March 9 was very smooth with only minor and momentary boiling instabilities.

Sodium and potassium were distilled from the loop components and samples were taken from each circuit during the draining operation. The results of the analyses performed on the alkali metal samples indicated that no contamination had occurred during the experiment.

Calibration of the slack diaphragm pressure transducers indicated that no changes had occurred in the response characteristics of the transducers during the 5,000-hour test.

Visual examination of the test loop proper and the auxiliary components gave no indication of system degradation or contamination. In most cases the appearance of the refractory alloy components was nearly identical with the pre-test appearance.

The Mo-TZM alloy turbine simulator nozzle and blade specimens showed no significant corrosion or erosion. Weight change data obtained on the nozzles and blades indicated that no changes had occurred during the test.

Preliminary chemical analysis results on Cb-1Zr foil and tube specimens indicated only very slight oxygen contamination resulted from exposure in the vacuum environment during the 5,000-hour test.

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III. PROGRAM STATUS

A. RESUMPTION OF PROTOTYPE CORROSION LOOP OPERATION

The Prototype Loop test was restarted on January 17, 1966, after repairs to the water cooling channel of the spool section were completed[1]. The start-up procedure is given here in detail as it departs from the procedure used in the initial startup which was described in an earlier report[2]. A change in the start-up procedure was made so that it would not be necessary to move the metering valve plug from the set position. Although the valve could have been moved and returned to the approximate test position, it was felt that even a small change in the position of the plug could affect the operating conditions of the loop and obscure any small changes in the loop operating characteristics which could have occurred during the loop shutdown.

The 500°F chamber bakeout was limited to the bell jar since the epoxy resin used in sealing the water cooling channels on the spool section[3] was limited to an operating temperature of 250°F. A bell jar temperature of 500°F was sufficient to heat the entire loop including the surge tank above the melting point of the liquid metal. The spool section reached a maximum temperature of 245°F during the bakeout and a vacuum chamber pressure of 1.5×10^{-7} torr was obtained at the end of the 40-hour bakeout period. The sodium and potassium EM pumps were turned on and set at 10% of the rated power to heat the pump ducts to approximately 500°F and outgas the metal foil insulation which surrounds the duct.

Prior to filling the sodium circuit, the EM pump was turned off. The argon pressure in the sodium surge tank was then increased from 0 psia to 20 psia to fill the loop. The pump was restarted and when sodium circulation was established the sodium heater was turned on and set at 1.5 kw. The secondary loop was then filled in the same manner by increasing the argon pressure in the potassium surge tank from 0 to 7 psia. The potassium pump was then set at 25% of the 0.11 GPM design flow rate and all liquid operation at a maximum temperature of 1000°F was continued for the next 14 hours to outgas the loop. The vacuum chamber pressure decreased from 6×10^{-7} to 1.5×10^{-7} torr during this period.

Boiling in the potassium circuit was initiated by slowly increasing the power to the sodium heater. Boiling occurs when the potassium temperature exceeds the saturation temperature as set by the argon pressure in the surge tank. At 0853 hours on January 18, 1966, boiling operations began at a sodium inlet temperature of 1295°F and a potassium vapor temperature of 1290°F. Boiling was first detected by pressure oscillations of less than ± 1 psi at the boiler outlet. A decrease in the rate of temperature rise of the sodium circuit was also observed as more heat was transferred from the sodium circuit to the boiling potassium. During this rise in loop operating temperature, the vacuum chamber pressure increased to 2.6×10^{-6} torr.

During the next seven hours, the loop was slowly returned to the operating conditions which prevailed prior to the shutdown. The rate of the temperature rise was governed by the outgassing of the loop components as they increased in temperature. Loop control during this period was smooth and normal. Small adjustments to the power and flow rate were made as the chamber pressure decreased into the 10^{-7} torr range. The procedure was to increase the heater power and then increase the secondary flow rate; using this technique, the boiling could be contained in the plug. If the boiling moved out of the plug, a temporary instability occurred which could be corrected by either decreasing the potassium flow rate or increasing the heater power, provided that the pressure in the test chamber was sufficiently low. During this stepwise procedure, the power to the preheater was also increased to raise the potassium inlet temperature.

A leak check of the vacuum system during the startup revealed a small leak in the main flange between the spool and the bell jar which was attributed to differential thermal expansion between the two flanges as the heat load varied in the chamber. The flange was resealed by retorquing the bolts to their pre-bakeout values.

The new start-up procedure was smoother and less complicated than the original startup and is recommended for future tests of this type. A comparison of the loop operating conditions before the 3,809-hour shutdown and after resuming the test indicated no significant differences in operating conditions.

The loop conditions just prior to the end of the 5,000-hour test are shown in Figure 1 and are typical of the conditions before and after the 3,809-hour test shutdown.

B. ARGON INSTABILITIES IN THE GETTER-ION PUMP

So-called "argon instabilities" have occurred in the Prototype Corrosion Loop test chamber getter-ion pump system at fairly regular time intervals of 50 to 70 hours since the beginning of the test. A complete record of each of these pressure surges has been reported in the quarterly progress reports[4,5]. A typical pressure surge of the type observed in the Prototype Loop test chamber consists of a very rapid rise (less than 5 seconds) from a base pressure of 2.5×10^{-8} to 1.5×10^{-6} torr and a more sluggish (approximately 5 minutes) return to the pre-surge pressure level. Pressure bursts of this type are frequently encountered in diode type getter-ion pumps[6]. The referenced report states that it is typically necessary to pump air at 1×10^{-5} torr for several hundred hours or 1×10^{-6} torr for several thousand hours before argon instabilities are observed. During both the Component Evaluation Loop II test and the Prototype Corrosion Loop test "argon instabilities" were encountered after several hundred hours of operation at indicated ion gauge pressures in the 10^{-7} to 10^{-8} torr range[6]. Although these instabilities have been studied for sometime, Jepsen, et al.[6], report that no completely satisfactory explanation for the instable argon behavior has been proposed. In another study of the argon instability phenomena, Brubaker[7] has reported
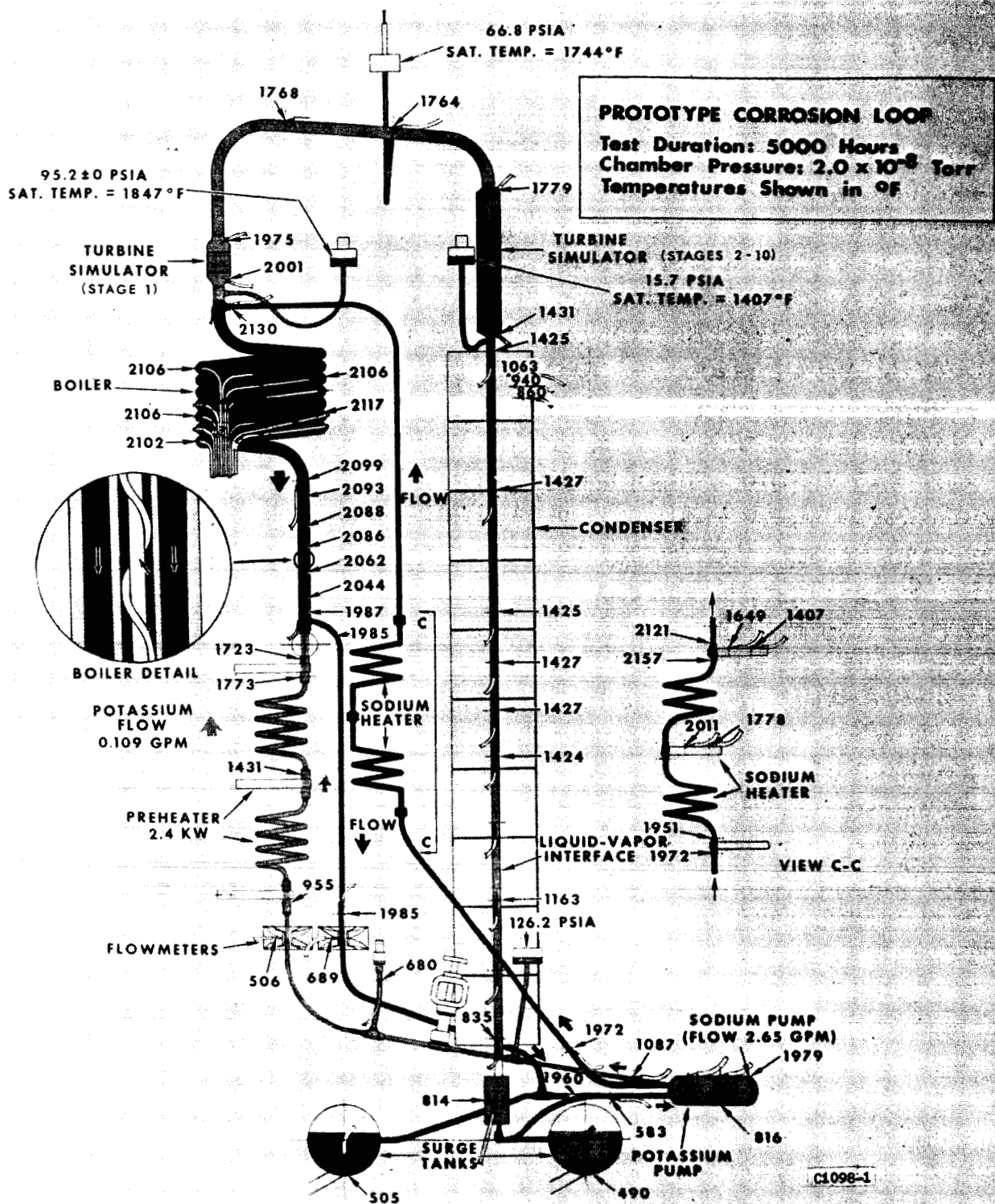
Figure 1. Prototype Corrosion Loop Test Conditions. (CDC 5458)

that none of the other gages present are found to partake in the violent pressure excursions experienced by the argon. In order to determine if the pressure surges observed in the test chambers at General Electric were due solely to argon, continuous scans for individual species were conducted. An argon peak height was measured during an instability which occurred on January 28, 1966, and the hydrogen peak height was measured during an instability which occurred on February 2, 1966. It was somewhat of a surprise to note that the surge in hydrogen pressure during the instability was somewhat larger than the argon pressure surge. These results are summarized in Table I. Since both argon and hydrogen and possibly other gases, contribute to the observed pressure surges, these results do not agree with the results reported by Brubaker[7] above.

The instabilities reported here occur much less frequently than those usually cited in the literature and at a much lower argon pressure. For example, Brubaker[7] reports argon pressure instabilities at 1×10^{-5} torr argon pressure and at approximately 10 minute intervals while those found in the loop test chambers occurred at argon pressures near 5×10^{-9} torr and at 50- to 70-hour intervals. These observations substantiate the rather general rule that the frequency of the instabilities is roughly proportional to the argon pressure. Thus while the instabilities are noted at rather long time intervals and are not detrimental to Prototype Test Loop operation, they can complicate the operation of pressure sensing systems designed to shutdown tests of this type when pressure rises occur. Following completion of the 5,000-hour test, consideration will be given to replacing the standard getter-ion pump elements with slotted "Super" getter-ion pump elements* which have an argon pumping speed of 6 percent of the air speed compared to 1 percent for the standard elements.

C. TERMINATION OF THE PROTOTYPE CORROSION LOOP TEST

The Prototype Corrosion Loop Test was terminated at 0700 on March 9, 1966 after completing 5,000-hours of operation. The power to the heaters and pumps was gradually reduced over a one-hour period with no significant pressure or temperature instabilities occurring during the shutdown. While the loop was cooling down, tests were made to determine the ease of operation of the metering and isolation valves in the potassium circuit and the shutter assembly on the condenser-subcooler. Both valves were opened and closed approximately one-half the total valve stem travel (0.125 inch). Operation of the valves was very smooth for the first 20 or 30 mils of stem travel but became rough with additional opening. The roughness is attributed to galling between the stainless steel drive gears of the valves. (A material with less galling tendencies than stainless steel will be used in fabricating drive gears for valves in future tests.) The shutter assembly was opened and closed three times and required very little torque on the magnetic rotary feedthrough. When the loop had cooled to less than 800°F, the argon was pumped from the surge tanks and the two circuits drained to these tanks.

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* Varian Vacuum Division, Palo Alto, California.

TABLE I.   ARGON AND HYDROGEN PRESSURE SURGES DURING "ARGON INSTABILITIES" IN THE PROTOTYPE CORROSION LOOP TEST CHAMBER

	Argon Scan 1-28-66 (1520)	Hydrogen Scan 2-2-66 (1430)
Indicated Ion Gauge Pressure	3.0×10^{-8} torr	2.5×10^{-8} torr
True Base Pressure[a] of Gas Prior to Instability	4.3×10^{-9} torr	7.5×10^{-9} torr
True Peak Pressure of Gas During Instability	2.0×10^{-8} torr	$> 3.3 \times 10^{-8}$ torr*
True Pressure 10 Seconds Later	9.0×10^{-9} torr	1.4×10^{-8} torr

*Peak was off scale on the 10×10^{-10} amp scale.

[a] True Pressure (Torr) is obtained by dividing the Ion Gauge Partial Pressure (Torr) by the ion gauge sensitivity relative to nitrogen.

Pressure rise rates were obtained with the loop at operating temperature and with the system at room temperature following test shutdown. Following test shutdown and before opening the test chamber, the partial pressure gas analyzer was recalibrated using the gases, helium, hydrogen, nitrogen and argon. The results of this calibration and a discussion of the test chamber environment and pressure rise rates are discussed in Section D of this report.

The test chamber was opened on March 11, 1966, and the components examined visually by the NASA Technical Managers and program personnel. In general, the pretest and post-test appearance of the visible components was quite similar. Two views of the loop following removal of the bell jar are shown in Figures 2 and 3.

D. TEST CHAMBER ENVIRONMENT

During operation of the Prototype Corrosion Loop, residual gas analyses were performed using a GE magnetic deflection type partial pressure analyzer. The analyzer tube (Model 22PT120) has an exposed ion source and is mounted on the chamber sump as shown in Figure 4. The system ion gauge, against which the partial pressure analyzer is calibrated, and the variable leak valve also is shown in Figure 4. The variable leak valve is used to admit various gases into the chamber during calibration of the analyzer. Figure 5 shows the electronic control unit (GE Model 22PC120) for the partial pressure analyzer, and the X-Y recorder used to record the mass spectra.

1. Calibration of the Partial Pressure Analyzer

The partial pressure analyzer was initially calibrated before the loop was installed in the vacuum chamber. This calibration, described in a previous progress report[8], showed excellent sensitivity of the analyzer. However, after the loop was installed in the chamber and the chamber once again evacuated and baked out, the sensitivity was quite low compared to that obtained in the initial calibration. This loss of sensitivity has been generally observed when the electron multiplier is exposed to the atmosphere. The electron multiplier was then baked out at about 600°F for 20 hours with a resulting increase in sensitivity to about 5% of its previous level.

After the loop was installed in the chamber, there was no opportunity to perform an accurate calibration of the analyzer because of the relatively high pressure (10^{-7} torr range) within the chamber. For the calculation of the various partial pressures during loop operation, it was thus necessary to indirectly obtain the required calibration factors. The calibration factor is here defined as that number by which the ion current is multiplied to give the indicated ion gauge partial pressure of a particular gas. The general procedure used in obtaining these factors was to assume that the relative sensitivity from one gas to another was the same as for the initial calibration, and that the sum of the calculated partial ion gauge pressures is equal to the ion gauge reading. A summary of the calibration factors used throughout the Prototype Loop tests is given in Table II. The third column
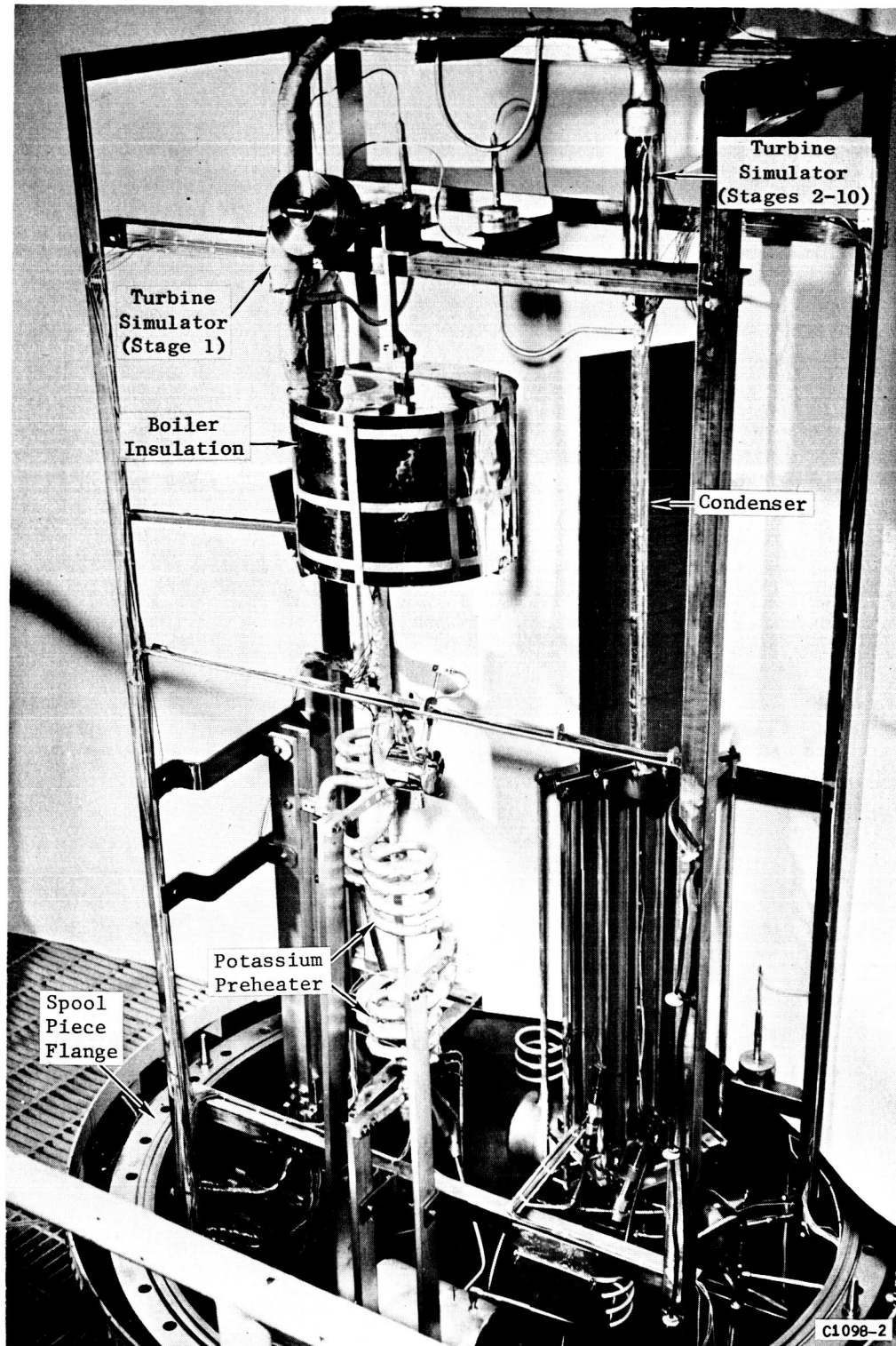
Figure 2.   Prototype Corrosion Loop After Removal of the Vacuum Chamber
Bell Jar Following Completion of the 5000-Hour Test.
(Orig. C66031427)
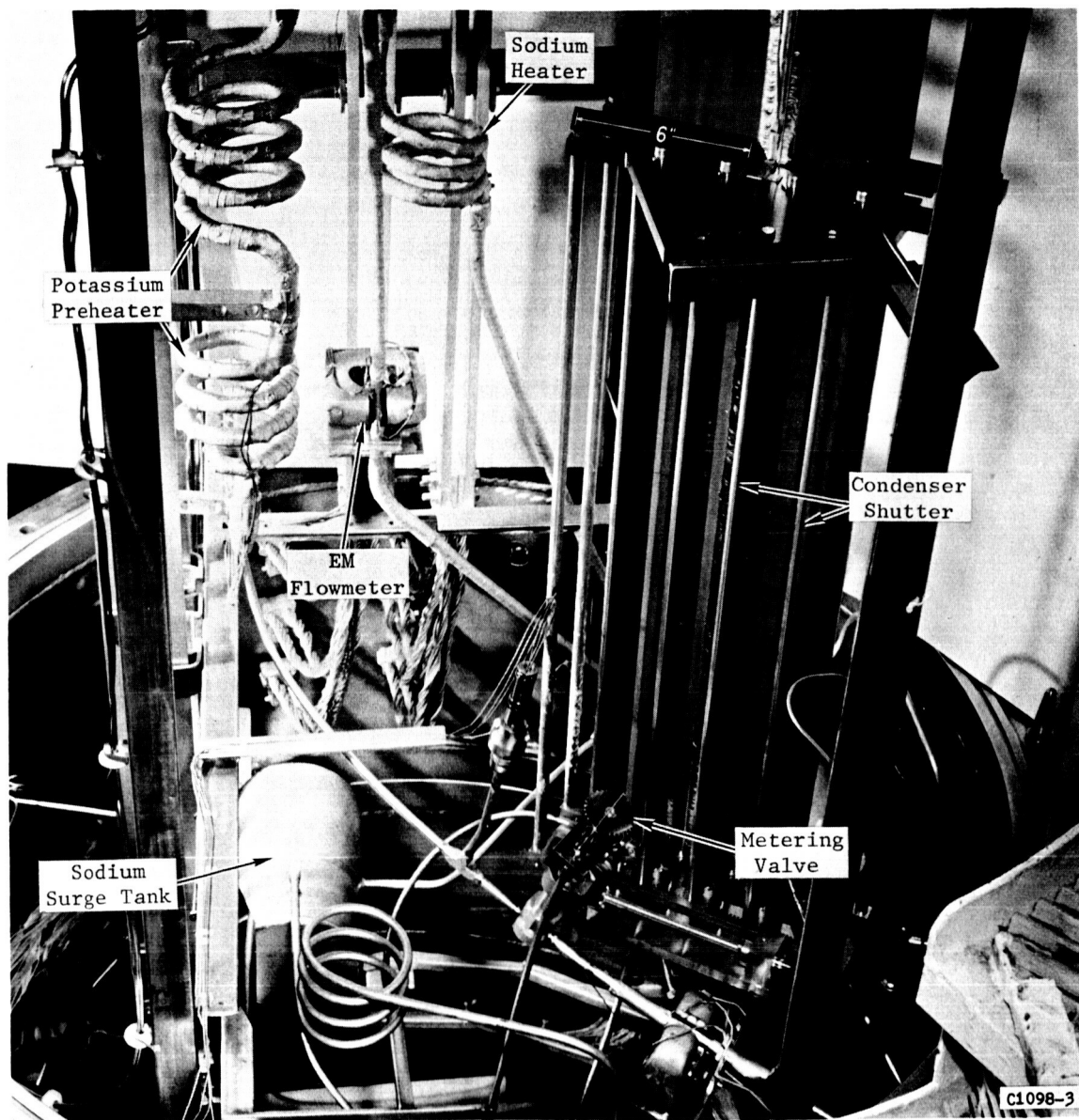
Figure 3. Lower Portion of the Prototype Corrosion Loop Following Removal of the Vacuum Chamber Bell Jar. (Orig. C66031432)
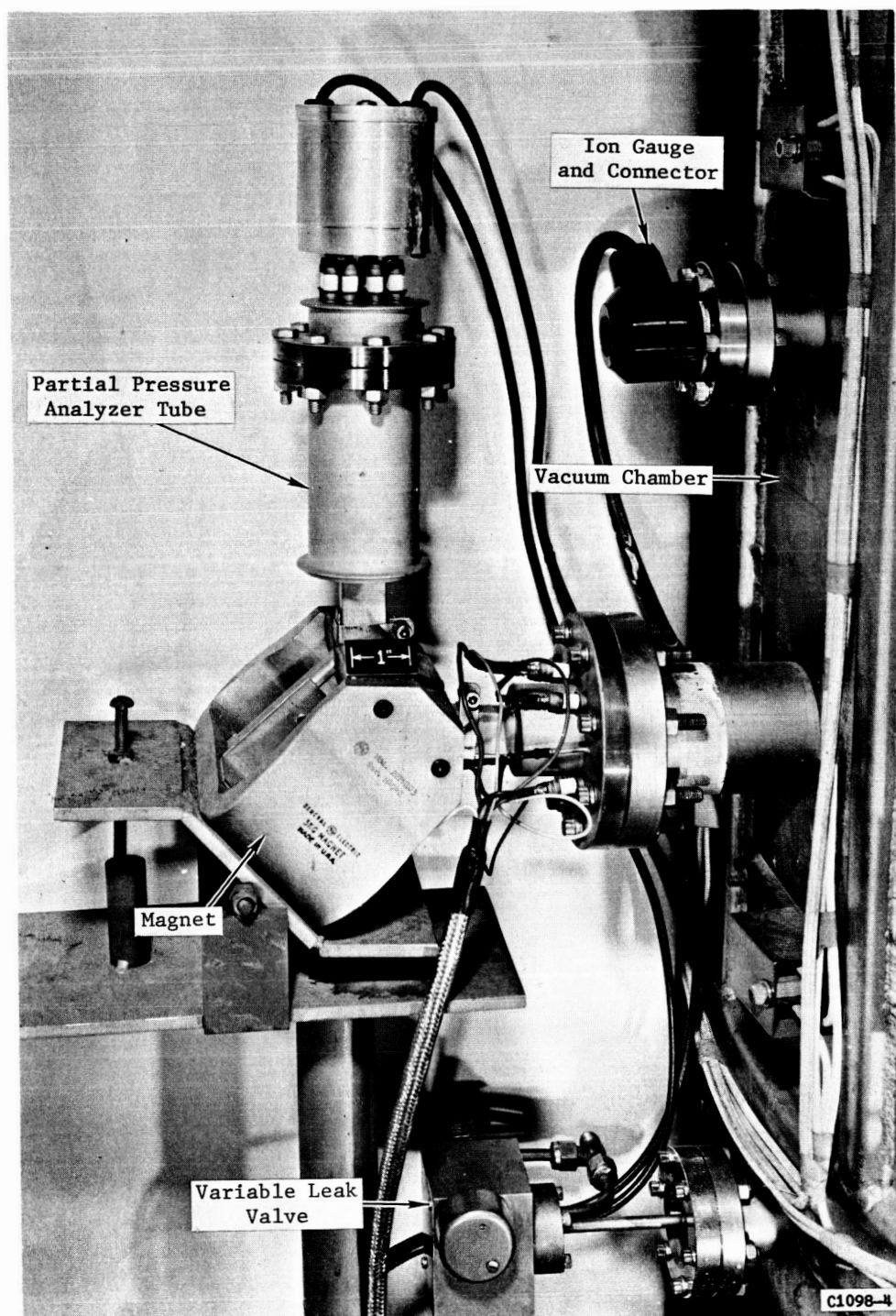
Figure 4. Ion Gauge, Partial Pressure Analyzer Tube and Variable Leak Valve
Mounted on Chamber Sump. (Orig. C66050427)
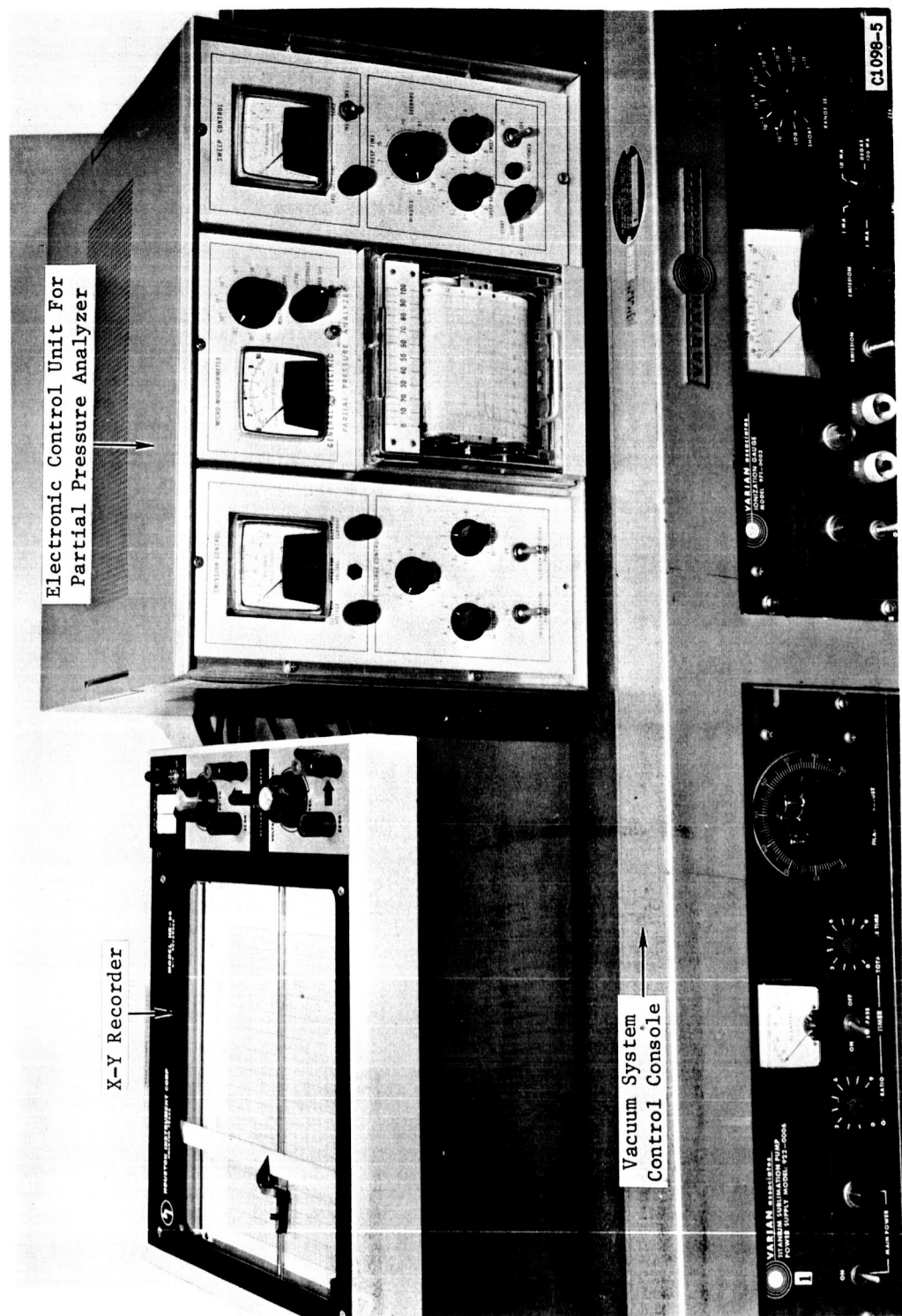
Figure 5. Electronic Control Unit and X-Y Recorder for the Partial Pressure Analyzer. (Orig. C66050426)

TABLE II.   PROTOTYPE LOOP RESIDUAL GAS ANALYSIS CALIBRATION FACTORS

Parent Specie	m/e*	Calibration Factor, K_i, torr/amp				Relative Ion Gauge Sensitivity
		Initial Calibration 3-3-65	-77 to +187 Test Hours	+187 to 3,809 Test Hours	3,809 to 5,000 Test Hours	
H_2	2	0.0138	0.25	0.28	14	0.42
He	4	0.059	0.54	1.2	23	0.19
CH_4	16	0.048	1.4	0.97	19	1.07
H_2O	18	0.053	2.4	1.1	21	0.89
N_2 or CO	28	0.079	3.7	1.6	31	1.00
O_2	32	0.10	5.7	2.0	39	0.84
Ar	40	0.17	8.2	3.5	68	1.56
CO_2	44	0.27	16.4	5.5	105	1.37

* m/e = mass/charge.

of Table II shows the results of the initial calibration of the analyzer. Three separate sets of calibration factors were used throughout the duration of the Prototype Loop test. The first set of factors, shown in column 4 of Table II, was used for spectra obtained between -77 and +187 test hours. At 187 test hours, the deflection electrode voltages were readjusted and new calibration factors were calculated. These factors, shown in column 5 of Table II, were used for spectra obtained between 187 and 3,809 test hours. During the test shutdown at 3,809 test hours, a new analyzer tube was installed and used until the conclusion of the test. This change in analyzer tubes necessitated another evaluation of the calibration factors for the different species, as given in the sixth column of Table II. At the conclusion of the 5,000-hour test, an attempt was made to calibrate the analyzer tube. Difficulties were encountered in this calibration due to the relatively high pressure (10^{-8} torr range) in the chamber and inability to hold constant pressure when a pure gas was admitted to the chamber. Furthermore, addition of one gas to the chamber resulted in considerable increase in the pressures of the other gases. In spite of these problems, calibration factors within about 20% of the values shown in column 6 of Table II were obtained for H_2, He, N_2 and Ar.

In the last column of Table II, the relative ion gauge sensitivities for the various species are listed. True partial pressure of a particular gas may be obtained by dividing the indicated ion gauge partial pressure by the relative ion gauge sensitivity.

2. Interpretation of Mass Spectra

Since complete cracking patterns were not obtained for all gaseous species, it was not possible to completely determine the contribution of each ion at every mass number. However, for most of the test duration, chamber pressures were in the low 10^{-8} torr range and mass spectra were simple enough that there was little difficulty in calculating the partial pressures of the individual species.

A typical mass spectrum, obtained after 2,494 hours of loop operation, is shown in Table III. The second column of Table III gives the ion with the greatest contribution at a particular mass number. The peak at m/e = 14 could possibly be due to CH_2^+, CO^{++}, or N^+. However, the magnitude of the 14 peak in comparison to the 12 and 15 peak indicates that the major contribution is from N^+. Similarly, the major contribution at m/e = 28 is from N_2^+. The magnitude of the 15 peak in comparison to the 32 peak indicates that the peak m/e = 16 contains contributions from both O^+ and CH_4^+. The ion current at m/e = 20 is attributed to Ar^{++} rather than Ne^+ since no appreciable ion current was detected for the m/e = 22 neon isotope.

The ion gauge partial pressures, calculated from the calibration factors given in column 5 of Table II, are shown in column 4 of Table III. Although the pressure calculated from the fragment CH_3^+ peak at m/e = 15 does not represent a real residual gas pressure within the chamber, it is a relative indication of the total hydrocarbon content. The sum of the ion gauge partial

TABLE III. TYPICAL MASS SPECTRUM OBTAINED
DURING PROTOTYPE LOOP OPERATION

Run #175 - 11-14-65 (1000)

Ion Gauge: 2.55 x 10^{-8} Torr - 2,494 Test Hours

m/e	Major Ion	Ion Current (Amp)	Ion Gauge Partial Pressure[a] (Torr)	True Partial Pressure[b] (Torr)
2	H_2^+	7.9 x 10^{-9}	2.2 x 10^{-9}	5.2 x 10^{-9}
4	He^+	1.0 x 10^{-11}	1.2 x 10^{-11}	6.3 x 10^{-11}
12	C^+	3.1 x 10^{-11}		
13	CH^+	0.5 x 10^{-11}		
14	N^+	6.0 x 10^{-10}		
15	CH_3^+	0.7 x 10^{-10}	0.7 x 10^{-10}	0.7 x 10^{-10}
16	O^+, CH_4^+	1.9 x 10^{-10}		
17	OH^+	1.9 x 10^{-10}		
18	H_2O^+	5.0 x 10^{-10}	5.4 x 10^{-10}	6.1 x 10^{-10}
20	Ar^{++}	5.0 x 10^{-10}		
28	N_2^+	5.1 x 10^{-9}	8.2 x 10^{-9}	8.2 x 10^{-9}
32	O_2^+	1.1 x 10^{-10}	2.2 x 10^{-10}	2.6 x 10^{-10}
40	Ar^+	2.9 x 10^{-9}	1.02 x 10^{-8}	6.5 x 10^{-9}
44	CO_2^+	1.2 x 10^{-10}	6.6 x 10^{-10}	4.8 x 10^{-10}
	T O T A L		2.21 x 10^{-8}	2.14 x 10^{-8}

a) Ion Gauge Partial Pressure (Torr) is the product of the mass spectrometer ion current (amp) and the calibration factor (Torr/amp) obtained for the gas.

b) True Partial Pressure (Torr) is obtained by dividing the Ion Gauge Partial Pressure (Torr) by the ion gauge sensitivity relative to nitrogen.

pressures is 13% lower than the actual ion gauge reading. This is considered to be quite good agreement, considering the approximations involved in the calibration. The fifth column of Table III gives the true partial pressures of the various species. It should once again be noted that the ion gauge partial pressures are obtained directly from the calibration of the partial pressure analyzer against the ion gauge. These values are sometimes referred to as "equivalent nitrogen pressures." The true partial pressures of the various gases are obtained from the ion gauge partial pressures and the relative ion gauge sensitivities which thus account for the differences in ionization efficiencies of the various gases. The main effect on the major species in the spectrum shown in Table II is that the true hydrogen partial pressure is considerably greater than the ion gauge partial pressure of hydrogen, while the true argon partial pressure is lower than the ion gauge argon pressure. The true nitrogen partial pressure is, of course, the same as the ion gauge partial nitrogen pressure since the ion gauge is calibrated for nitrogen.

3. Partial Pressures of Various Species During Loop Operation

Analyses of the residual gases in the chamber were performed at least once each day during the 5,000-hours of operation of the Prototype Loop. During the two start-up periods, analyses were performed more frequently in order to observe variations in partial pressures as the different components reached their operating temperatures. In Figure 6 are shown the ion gauge reading and the true partial pressures during the 5,000-hours of Prototype Loop operation. Data are plotted at approximately 50-hour intervals. The ion gauge reading remained below 1×10^{-7} torr after 130 hours of loop operation and was between 2×10^{-8} and 3×10^{-8} torr for most of the test. The major gaseous species in the system were N_2^+, Ar^+, and H_2^+. The peak at $m/e = 28$ contains contributions from both N_2^+ and CO^+, with the greater contribution from N_2^+, as discussed above. In a typical mass spectrum such as shown in Table III, N_2^+, H_2^+, and Ar^+ account for 92% of the total pressure in the chamber. (It is interesting to note that a general review of all the mass analyzer traces obtained during the operation of 10^{-8} torr creep systems on a current NASA contract[9] indicated the following order of decreasing gas concentrations: CO^+, H_2O^+, CO_2^+, H_2^+ with small quantities of CH_4^+, Ar^+ and He^+.)

The relatively high nitrogen pressure in the Prototype Loop Chamber probably arose from air leakage into the chamber. The surprisingly high argon pressure through the duration of the test is also attributed to air leakage and to the relatively low pumping speed of the ion pumps for argon. The long term "average" pumping speed for argon appears to be about 2 percent of the nitrogen pumping speed[10]. Since the pumping speed for argon is lower than for nitrogen, the argon concentration builds up until equilibrium is reached where the quantity of argon pumped is equal to the quantity being admitted with the air. The result is that, under the particular conditions of loop operation, the argon pressure is only slightly less than the nitrogen pressure, even though the nitrogen to argon ratio in air is about 80. Hydrogen is evolved from various components in the system and is also invariably a major component in a well baked, ultra high vacuum system.

Of the minor constituents of the residual gases within the chamber, H_2O^+ and CO_2^+ are the most abundant. Since these substances are strongly adsorbed on internal surfaces, their presence can generally be detected even in a very thoroughly baked ultra high vacuum system. Oxygen pressure was below 1×10^{-9}

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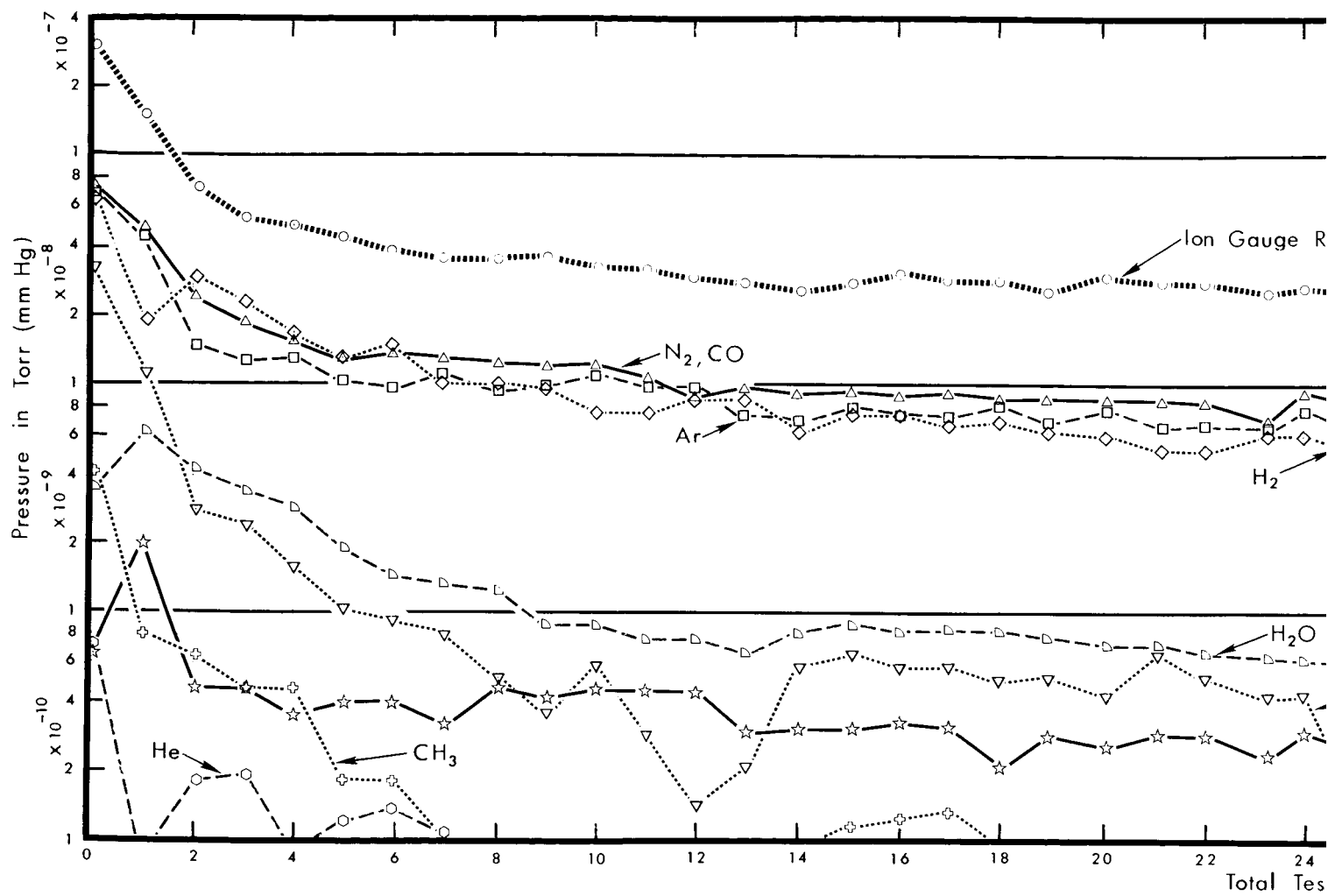
Figure 6.   Vacuum Chamber
            Loop Test. (C6...

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Symbol	Gas
○	Ion Gauge Reading
◇	H_2
○	He
⊕	CH_3
◁	H_2O
△	N_2, CO
☆	O_2
□	Ar
▽	CO_2

Test Restarted

Test Shut Down Because
of Pressure Rise

eading

CO_2

O_2

26 28 30 32 34 36 38 40 42 44 46 48 50

Hours X 10^{-2}

C1098-6
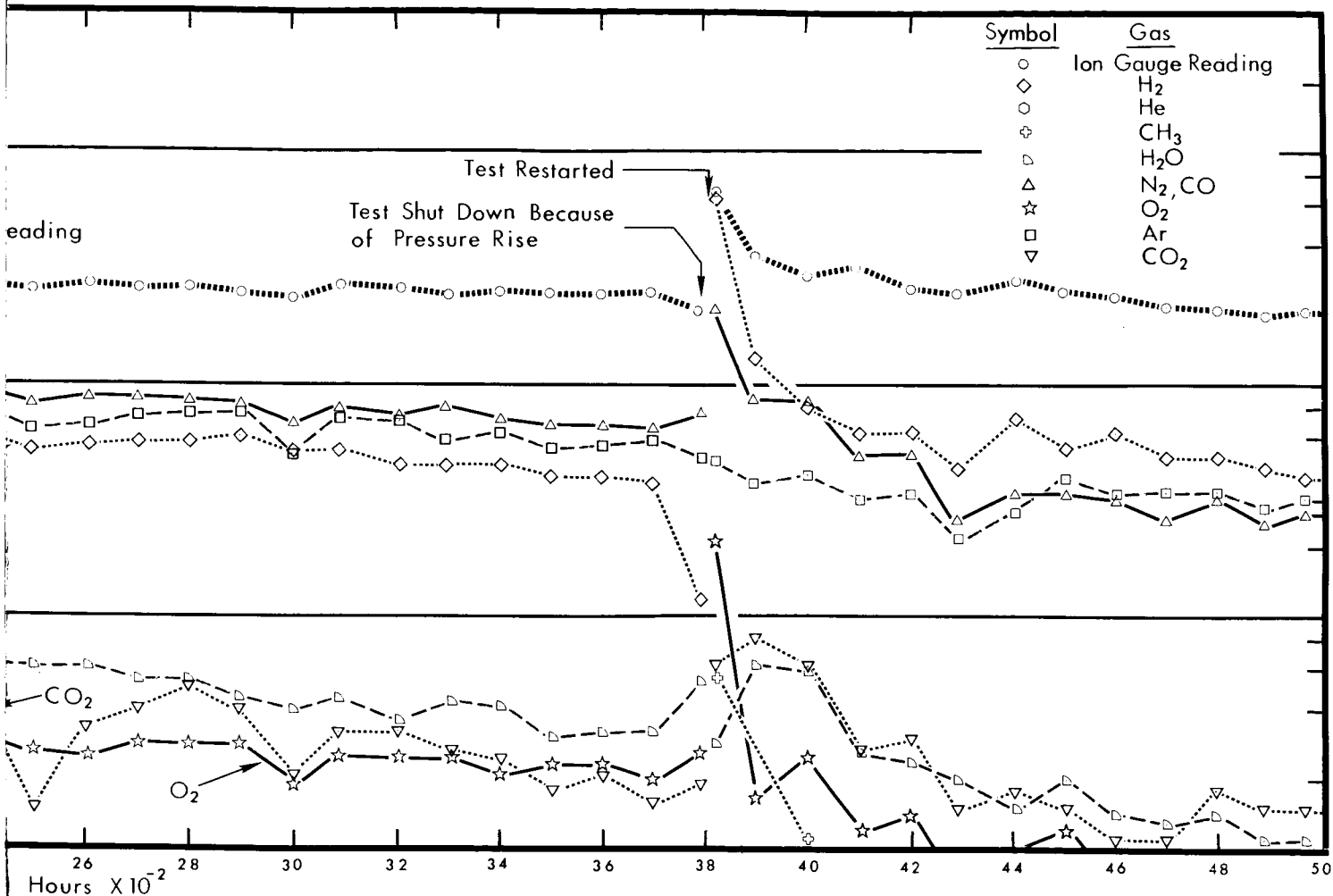
Pressures During the 5000-Hour Prototype Corrosion
(052351)

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20

torr for most of the test and was typically about 3×10^{-10} torr. The main source of oxygen appears to be air leakage. However, the nitrogen to oxygen ratio is about 30 compared to the value of about 4 for air even though oxygen pumping speed of the ion pumps is only about 60 percent of the pumping speed for nitrogen[10]. It thus appears that there are additional mechanisms active in removing oxygen from the system. An obvious additional mechanism for removal of oxygen is reaction with the Cb-1Zr components at elevated temperature.

Figure 7 shows the variation in ion gauge reading and true partial pressures observed as the loop was brought up to operating temperature following the shutdown at 3,809 hours of loop operation. The main feature in this plot is the high hydrogen pressure observed during restarting of the loop. It should be noted that such high hydrogen pressure was not observed during the initial startup of the loop[11]. The fact that the hydrogen partial pressure calculated from the mass spectra actually exceeds the ion gauge reading for several points in Figure 7 does not necessarily represent an inconsistency since the reported true partial pressures have been corrected for ionization efficiency and no such correction has been applied to the ion gauge reading. It might also be noted in Figure 7 that the rather large pressure excursions experienced during restarting of the loop were accompanied quite generally by an increase in each of the partial pressures.

4. Chamber Pressure Rise Rate with Ion Pumps Off

At the conclusion of 5,000 hours of Prototype Loop operation, the ion pumps were turned off and the pressure rise rate within the chamber was noted over a period of one-half hour with the loop maintained at operating temperatures. A similar test was made after the loop was shutdown and allowed to cool for about 20 hours. At the conclusion of this cool down period, the loop components were essentially at ambient temperature. The results of these chamber pressure rise rate measurements with the ion pumps off is shown in Figure 8. With the loop at operating temperature, the pressure increased to 1.4×10^{-7} torr within 9 minutes followed by a more gradual rise to 2.5×10^{-7} torr at the end of a 30-minute period. With the loop at ambient temperature, an initial pressure rise to 9.2×10^{-8} torr in 6 minutes was followed by a more gradual increase to 2.5×10^{-7} torr after 35 minutes with the ion pumps off. The overall pressure rise rates are 7.0×10^{-9} torr/min with the loop at ambient temperature and 7.8×10^{-9} torr/min with the loop at operating temperatures.

Mass spectra of the residual gases in the chamber were obtained before and after the pressure rise measurements with the loop at ambient temperature. Partial pressures obtained from these spectra are shown in Table IV. These data show that 82% of the pressure increase is accounted for by the increase in argon pressure with appreciable increases in nitrogen and helium pressures. The increases in helium and argon are most probably due mainly to evolution of these gases from the pumps since the pressure rise rates are too high to be accounted for by air leakage and there seems to be no mechanism by which the pump could continue to remove the inert gases in the absence of an applied voltage. Concerning inert gas evolution from ion pumps, Rutherford
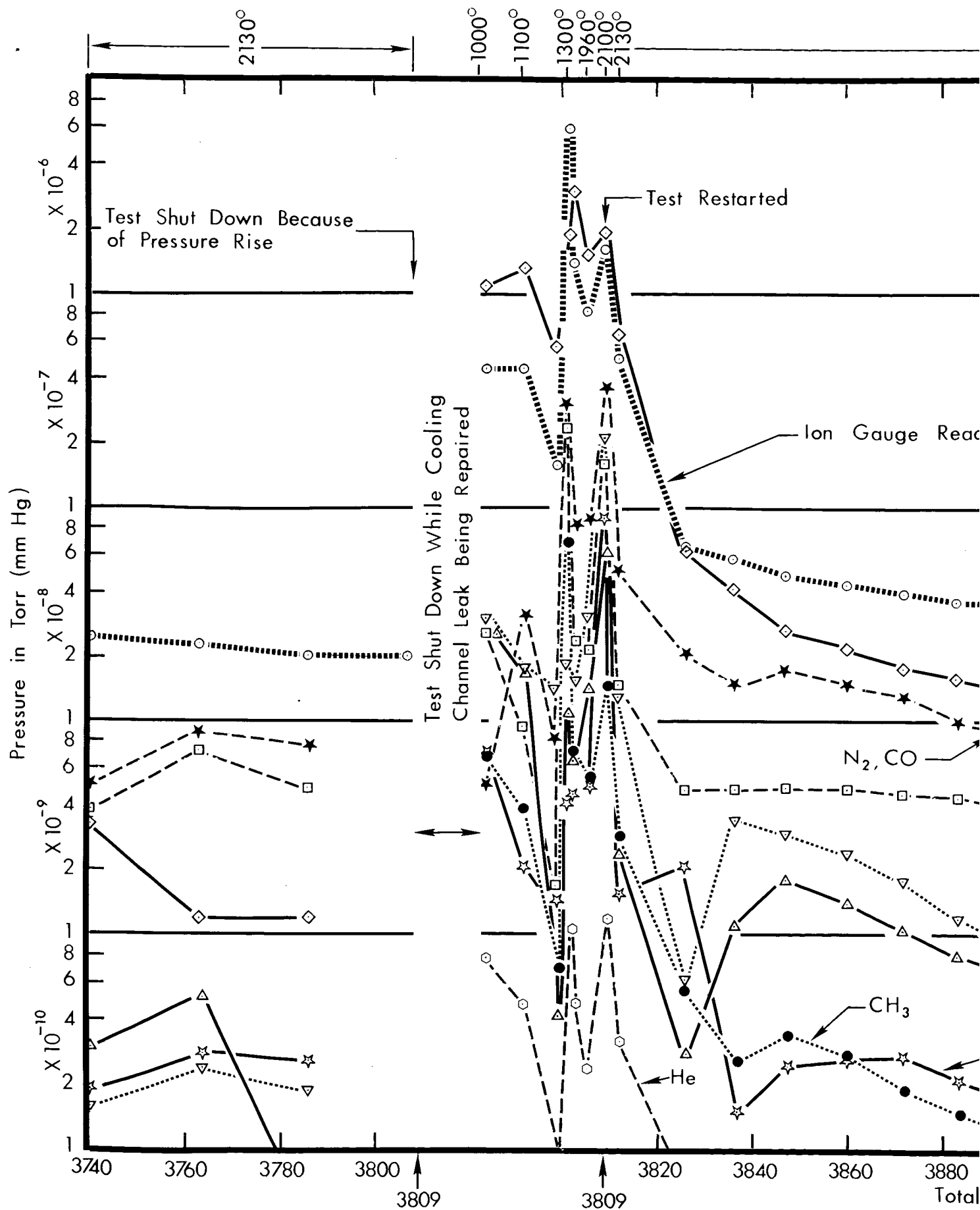
Figure 7. Vacuum Chamber Press
Shutdown at 3809 Hou

23

Symbol	Gas
○	Ion Gauge Reading
◇	H_2
⬡	He
●	CH_3
△	H_2O
✶	N_2, CO
✫	O_2
□	Ar
▽	CO_2

ling

H_2

Ar

CO_2

H_2O

O_2

3900 3920 3940 3960 3980 4000 4020 4040 4060 4080 4100

Test Hours
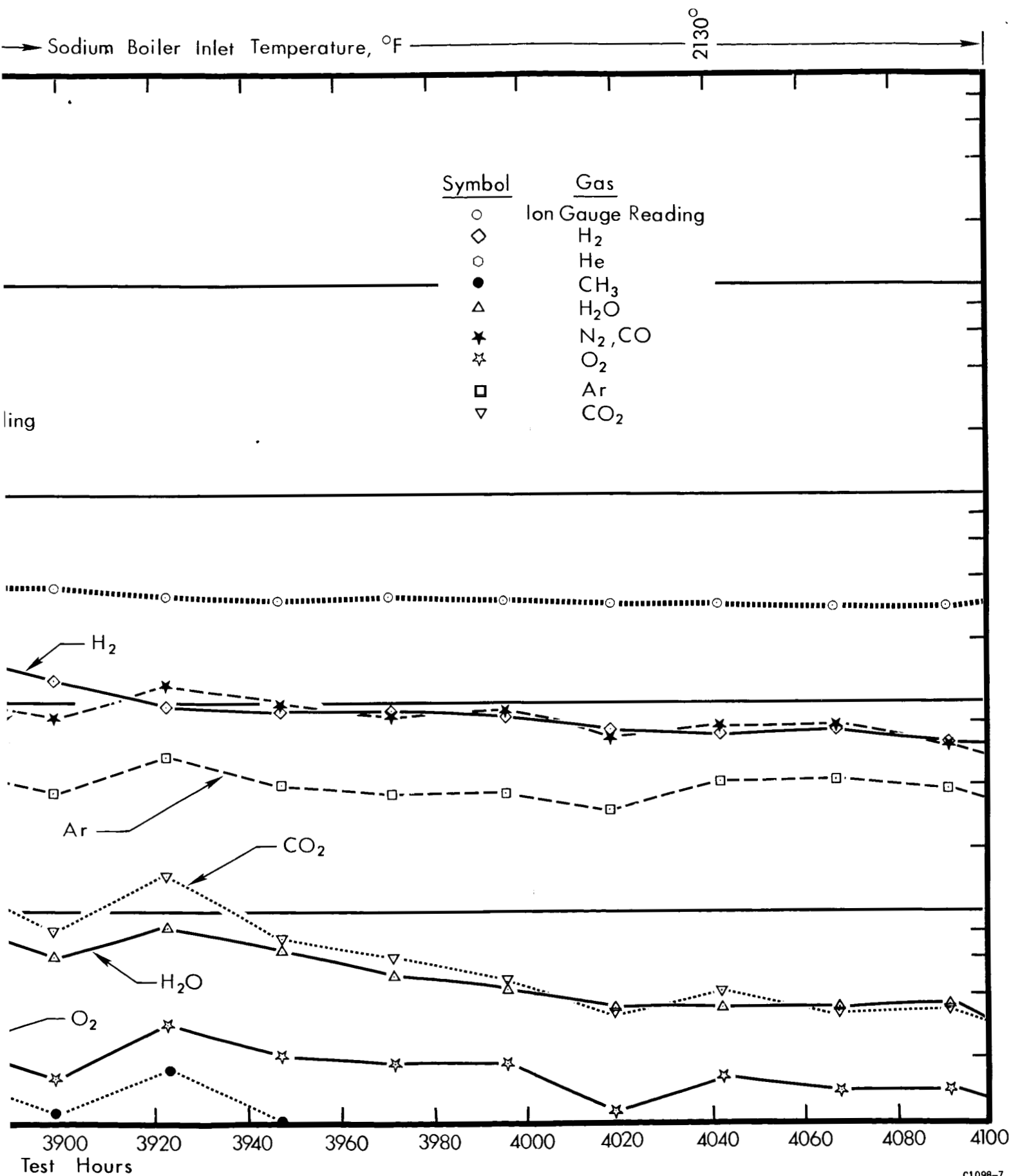
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ures During Startup and Operation Following
rs. (C6605235 2)

-23- ⟨24⟩
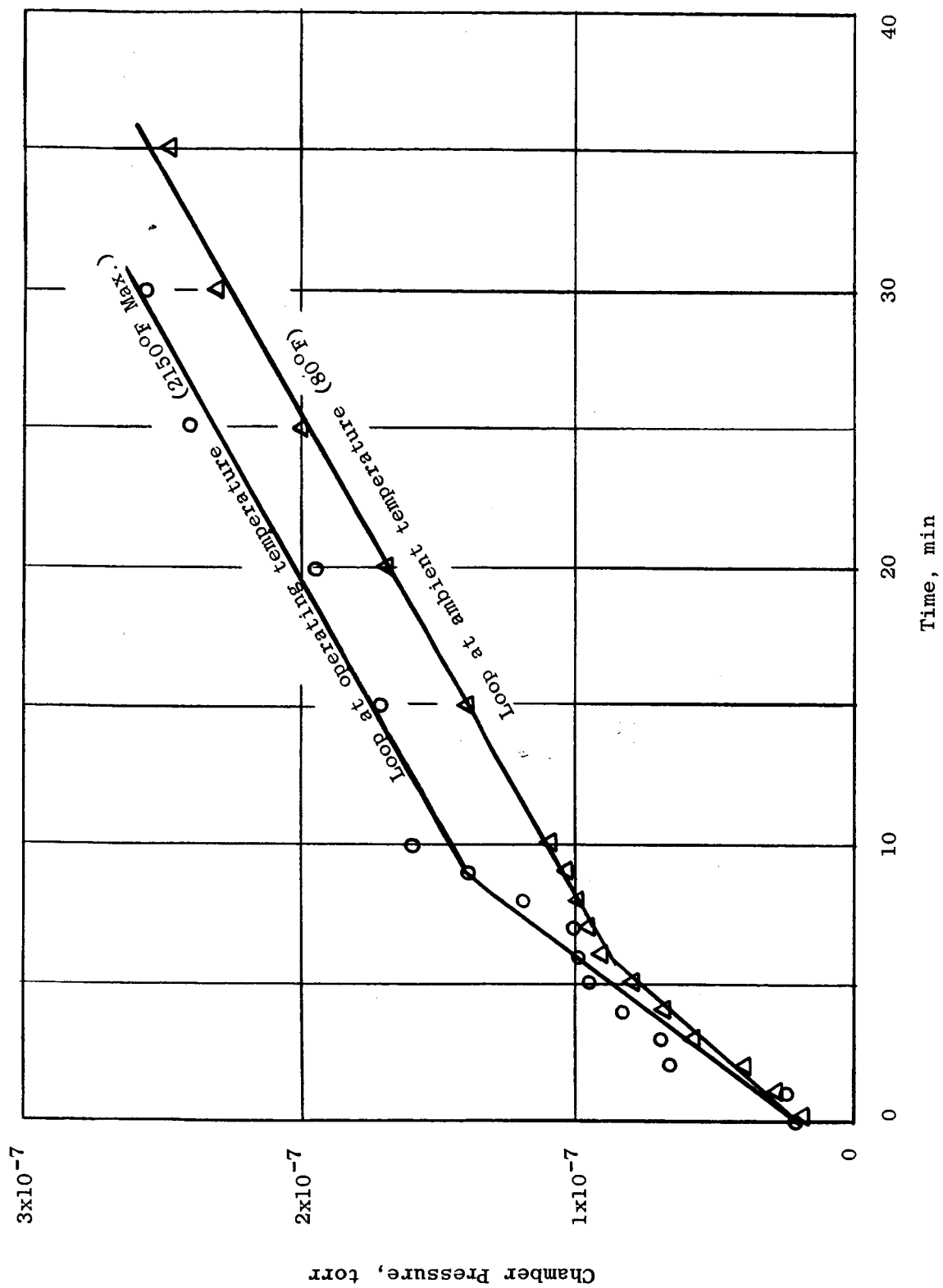
Figure 8. Chamber Pressure Rise Rate With Ion Pumps Off.

et al.[10] have stated that "argon re-evolution is thus of little concern during normal pump operation." On the basis of their argument, the statement should also apply when the pump is turned off. Roberts and Vanderslice[12], on the other hand, state that "atoms which have been driven into the surface and are not buried by sputtered metal can diffuse back into the gas phase."

The data of Table IV indicate that there is little increase in hydrogen pressure and no appreciable increase in oxygen pressure when the ion pumps are turned off for a 30-minute period with the loop at ambient temperature. Since there are certainly sources of these two gases, the small pressure increase indicates that there is some mechanism by which these gases are removed from the system even with no voltage applied to the ion pumps. It has, in fact, been reported[10] that when the discharge voltage is turned off, hydrogen pumping continues at a substantial rate for "some time."

One could assume also that a similar mechanism is effective in removing nitrogen from the system and thus the rate of nitrogen pressure increase is the net result of air leakage into the system and some continued pumping by the ion pumps. The increase in CH_3^+ is apparently due to the buildup of methane pressure within the system. The origin of this methane is not apparent.

In summary, it appears that when the ion pumps are turned off, they evolve previously pumped inert gases while continuing to pump the active gases at an appreciable rate, at least for a period of 30 minutes. If similar pressure rise rate measurements were continued over a long period of time, presumably the rate of increase of argon and helium pressures would diminish and the pressures of these two gases would approach a nearly constant value, while the pressures of the more active gases would increase at an accelerated rate as the pumping surfaces become saturated. It is, therefore, recommended that pressure rise rates be determined for periods of at least several hours in future tests of this type to obtain more information regarding the gas load and sources in the system.

E. DISTILLATION OF ALKALI METAL FROM THE LOOP CIRCUITS

There have been numerous cases reported by various alkali metal corrosion investigators of deleterious effects on test components resulting from interactions between alkali metals, stripping agents and the test components while removing residual alkali metal following completion of the corrosion experiment[13,14]. Quite often it is difficult to determine unequivocally if the observed "corrosion" occurred during the test or while removing the alkali metal with the stripping agent. This was a particularly critical problem in the Prototype Corrosion Loop in areas such as the turbine simulator where alkali metal would not drain from the many narrow annuli and where interactions with stripping agents, such as alcohol or butyl cellosolve, might negate the effort to obtain very accurate weight change data on the turbine simulator components. For these reasons, it was concluded that vacuum distillation of the alkali metals from the loop circuits to the surge tanks prior to draining and sampling these systems would be desirable.

TABLE IV.   RESIDUAL GAS ANALYSES BEFORE AND AFTER PRESSURE
RISE RATE MEASUREMENT WITH LOOP AT AMBIENT TEMPERATURE

m/e	Specie	Before Pressure Rise[a] Rate Measurement True Partial Pressure[c] Torr	After Pressure Rise[b] Rate Measurement True Partial Pressure[c] Torr
2	H_2	3.3×10^{-9}	3.8×10^{-9}
4	He	1.5×10^{-9}	2.9×10^{-8}
15	CH_3	---	5.6×10^{-9}
18	H_2O	---	---
28	N_2	5.5×10^{-9}	3.7×10^{-8}
32	O_2	4.6×10^{-10}	4.6×10^{-10}
40	Ar	2.8×10^{-9}	3.4×10^{-7}
44	CO_2	---	---
	TOTAL	1.4×10^{-8}	4.2×10^{-7}

(a) Ion pumps on Ion gauge reading - 1.35×10^{-8} torr.

(b) Ion pumps had been off for approximately 50 minutes. Ion gauge
reading - 3.5 to 4.2×10^{-7} torr.

(c) True Partial Pressure (Torr) is obtained by dividing the Ion Gauge
Partial Pressure by the ion gauge sensitivity relative to nitrogen.

The first step in the preparation of the loop for distillation of the residual alkali metal following completion of the 5,000-hour test was the removal of the thermal insulation shield assembly which surrounded and thermally insulated the boiler during test operation. The insulation was removed to expose the boiler coils directly to heat radiated from the distillation heaters and accelerate the distillation in the 244-inch long tube-in-tube assembly.

Specimens of the Cb-1Zr alloy foil were also taken from four regions of the loop, bagged and tagged for use in determining the extent of interstitual contamination that occurred in various regions during the 5,000-hour test. The foil samples were taken from the following locations:

    a) Nine-stage turbine simulator (single layer),

    b) Sodium line, 3 inches from boiler inlet (12 layers),

    c) Potassium vapor carryover line (12 layers) and

    d) Top coil of sodium heater, 4 inches from top electrode
        (12 layers).

The distillation heaters were six quartz infrared lamps* with tungsten filaments which were mounted on stainless steel brackets. These heater brackets were welded to the loop support structure as shown in Figure 9. The lamps were connected in parallel to the main heater power supply used in the loop operation. Two layers of stainless steel foil were wrapped around the loop support structure to thermally insulate the loop and quartz heaters from the test chamber bell jar as shown in Figure 10. Special precautions were taken in placing the quartz lamps so that the ends would protrude through the insulation out of the heated zone to reduce the possibility of failure of the end seals from overheating. The sodium and potassium surge tanks were also shielded from the quartz heaters to maintain a lower temperature than the loop so that the alkali metal distilled from the loop would condense in the cooler surge tanks located in the lower portion of the chamber.

After a low power electrical checkout of the heating lamps, the bell jar was lowered to the spool section and the vacuum chamber sealed. The turbo-molecular pump was used to rough pump the chamber to 5×10^{-4} torr. The ion pump was turned on and after approximately five hours the ion pump confined. The stainless steel foil thermal insulation which enclosed the loop not only contributed to the outgassing load but also reduced the conductance of the gases emanating from the surface of loop components. The chamber bakeout system was turned on after the chamber pressure was in the 10^{-7} torr range. The system was outgassed for approximately 50 hours with only the bakeout heaters on. The loop temperature during this period was approximately 500°F. The quartz heaters were then turned on at low power. The EM pump power was also turned on at approximately 10% of the rated power to heat-up the pump duct and to accelerate the outgassing of the metal foil insulation which

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* General Electric Type 3800 Te/VB, 3800 watts, 570 volts, 1/2-inch OD x 43-1/2-inch long.
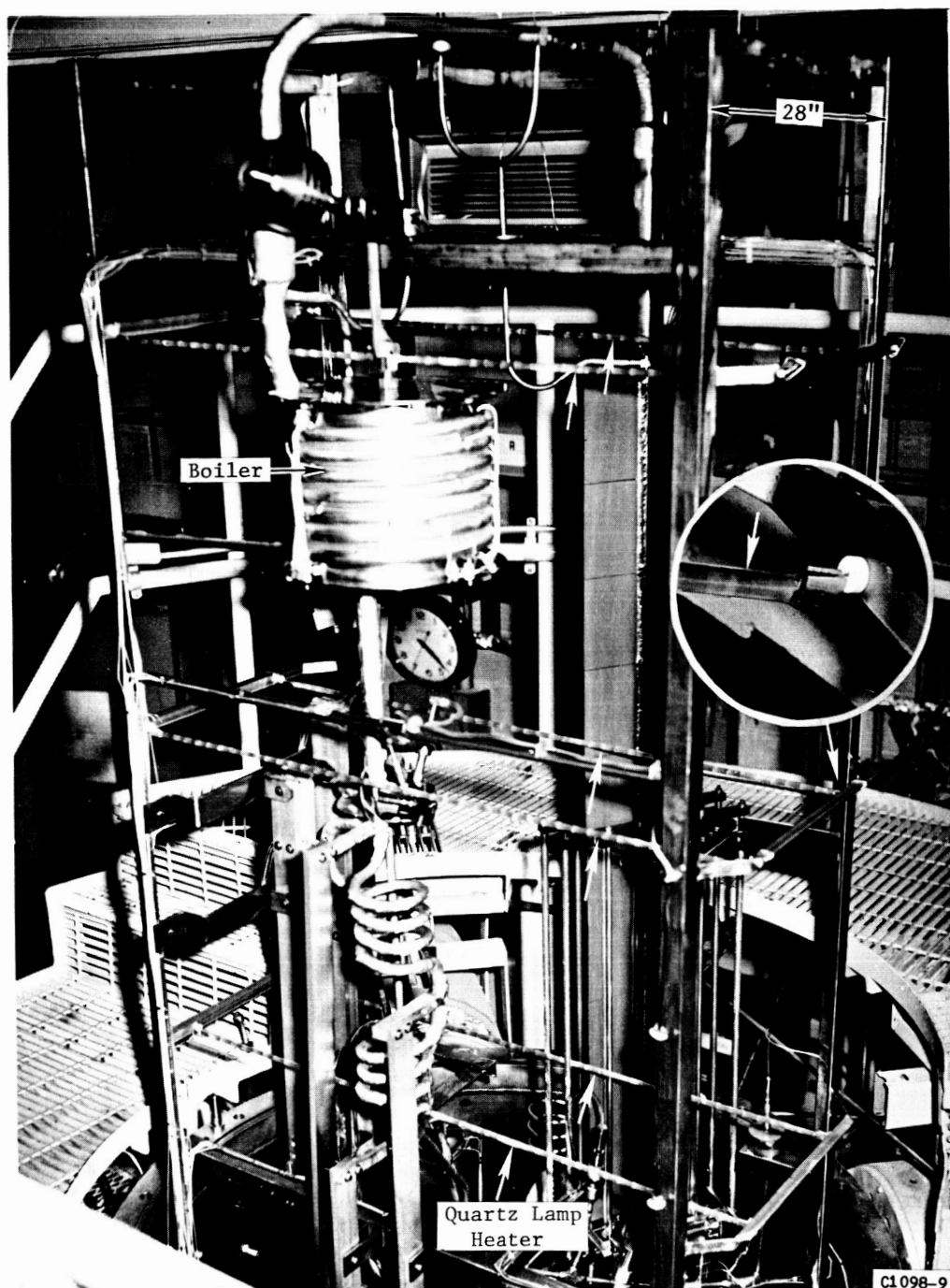
Figure 9. Prototype Corrosion Loop Following Installation of Quartz Lamp
Heaters for Distillation. Typical Heater Mount Shown in Enlarged
Insert. Six Quartz Heaters Indicated With Arrows. (Orig. C66031752,
C66031753)

Figure 10. Prototype Corrosion Loop Following Installation of Two Layers of
Stainless Steel Foil Reflective Insulation in Preparation for
Distillation of Alkali Metals From the Loop Circuits to the Surge
Tanks. (Orig. C66041410)

which surrounds the pump duct. During the next 30 hours, the quartz heaters
were slowly increased in power to reach the design distillation temperature
of 1000°F. The power was increased in small increments to hold the chamber
pressure in the 10^{-7} torr range. During this period the titanium sublimation
pump was used to assist the ion pump in handling the outgassing load.

At 1200 on April 2, 1966, the distillation period began with the loop
temperature at 960°F and the EM pump ducts at 1120°F. The vacuum chamber pres-
sure at this time was 4×10^{-7} torr. The stated thermal conditions were main-
tained for 100 hours and at the end of the distillation the chamber pressure
had decreased to 8.7×10^{-8} torr.

F. DRAINING AND SAMPLING OF ALKALI METALS FROM THE LOOP FOLLOWING DISTILLATION

At the end of the 100-hour distillation period described in Section E above,
the sodium and potassium were drained by gravity feed from the loop surge tanks
to the charge tanks located in the transfer systems. The transfer systems which
contain the charge tank, disposal tank and sampling system for each of the
alkali metals was described in an earlier report[15].

The sodium surge tank was gravity drained and subsequently pressurized
to 10 psia with high-purity argon to assure complete transfer of sodium to the
charge tank located in transfer system. Following the gravity drain of the
potassium surge tank, the Cb-1Zr alloy isolation valve located between the surge
tank and loop proper was closed and high-purity argon was used to pressurize
the potassium surge tank to 10 psia to complete the transfer of residual potas-
sium in the drain lines to the charge tank in the transfer system. During a
ten-minute period, no pressure rise was detected on the loop side of the isolation
valve indicating that a gas tight seal had been achieved when the isolation valve
was closed.

The potassium and sodium in the charge tanks of the transfer system were
sampled using the techniques described and illustrated in an earlier report[16].
The remaining potassium and sodium in the charge tanks was pressurized through
5-micron stainless steel filters to the disposal tanks. The draining, sampling
and filtering operations described briefly above are covered in detail in
Appendix A of this report.

The sodium and potassium samples obtained from the loop surge tanks follow-
ing completion of the test were analyzed for oxygen, nitrogen, total carbon and
metallic impurities. The results obtained from these analyses are presented in
Table V. As shown in the table, the oxygen concentrations as determined by the
amalgamation method for both sodium and potassium were at the 5 ppm level — the
same level found before the start of the endurance run. No nitrogen was detected
in either sample. The carbon concentrations obtained were about 20 ppm which
is considered a normal level for high purity sodium and potassium. Both alkali
metals contained metallic impurities at or near the limit of detection before
the test and the post-test results were essentially the same.

TABLE V. RESULTS OF ANALYSES OF SODIUM, POTASSIUM AND FILTER RESIDUES AFTER THE 5,000-HOUR ENDURANCE TEST OF THE PROTOTYPE LOOP

Sample Identity	Oxygen ppm	Nitrogen ppm	Carbon ppm	Al	Ag	B	Be	Ca	Cb	Co	Cr	Cu	Fe	Mg	Mn	Mo	Ni	Pb	Si	Sn	Ti	V	Zr	Na
													Metallic Elements, ppm in Alkali Metal											
Sodium From Primary Loop Surge Tank After 5,000-Hour Endurance Test, Sampled at 250°-300°F	5,6	<5 (ND)	18,26	<3	<3	<38	<3	<3	<13	<3	<3	<3	<3	<3	<3	<3	<3	<13	<3	<13	<3	<13	<13	--
Potassium From Secondary Loop Surge Tank After 5,000-Hour Endurance Test, Sampled at 250°-300°F	4,6	<5 (ND)	20,21	<2	<2	<19	<2	2	<10	<2	<2	<2	<2	<2	<2	<2	<2	<10	10	<10	<2	<10	<10	24
Residues From Five-Micron, Type 316 SS, Sintered Filters																								
Sodium	--	--	--	--	--	--	--	--	ND	--	D	--	D	--	--	ND	D	--	--	--	--	--	ND	--
Potassium	--	--	--	--	--	--	--	--	ND	--	D	--	D	--	--	ND	D	--	--	--	--	--	ND	--

D = Detected

ND = Not Detected

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G. CALIBRATION OF THE PROTOTYPE CORROSION LOOP PRESSURE TRANSDUCERS

The four slack diaphragm pressure transducers used in measuring the operating pressure of the potassium circuit of the Prototype Corrosion Loop were calibrated before and after the 5,000-hour test. The calibrations were made with the pressure transducers in the test position with the readout equipment used in the test operation. The pressure transducers were calibrated between 0-150 psia against a Wallace and Tiernan gauge calibrated to ± 0.1% of full scale. The millivolt output was measured by a recording potentiometer with an accuracy of ± 1/4% of full scale (50 millivolts).

The accuracy of the slack diaphragm pressure transducer as rated by the Taylor Instrument Company is ± 1/2% of the full range at 14.7 psia barometric pressure and room temperature. The effect of temperature on the output of the pressure transducer was estimated by the vendor to be 0.008 psi/°F at the sensing element and 0.002 psi/ft/°F around the capillary tube.

The pre-test calibration[17] was made with the potassium circuit filled and the vacuum chamber at 500°F which was the estimated operating temperature of the sensing element and capillary tube. The calibration after 5,000 hours of operation was made following the distillation treatment and draining operations described above. The transducers were calibrated at 950° and 600°F during the cool down period following the draining of the loop surge tanks. The higher temperature post-operation tests were possible because of the addition of infrared quartz heating lamps which were installed for distilling of the residual potassium from the loop before disassembly.

The results of the calibration before and after the 5,000-hour test are shown in Figure 11. No significant change in the accuracy of the pressure transducers were detected; the test data were within the ± 1/2% accuracy of the calibration system. Results obtained on all the transducers were similar.

The effect of temperature as estimated by the vendor was far too pessimistic. In fact, the difference in the calibration between the 600° and 950°F calibration could not be detected by the calibration equipment used. If the estimated temperature effect was correct, the 350°F change around the 15 feet long capillary tube would have resulted in an indicated pressure change of 10.5 psi which could easily have been detected with the calibrating equipment used.

The results of the calibration given in Figure 12, showed that no significant change in the performance of the pressure transducer was observed and that the ± 1/2% accuracy specified by the vendor can be attained. Although no significant effect of the temperature on the sensing element or capillary tube was observed, it is recommended that future calibration tests of this type be performed at or near the anticipated operating temperatures.
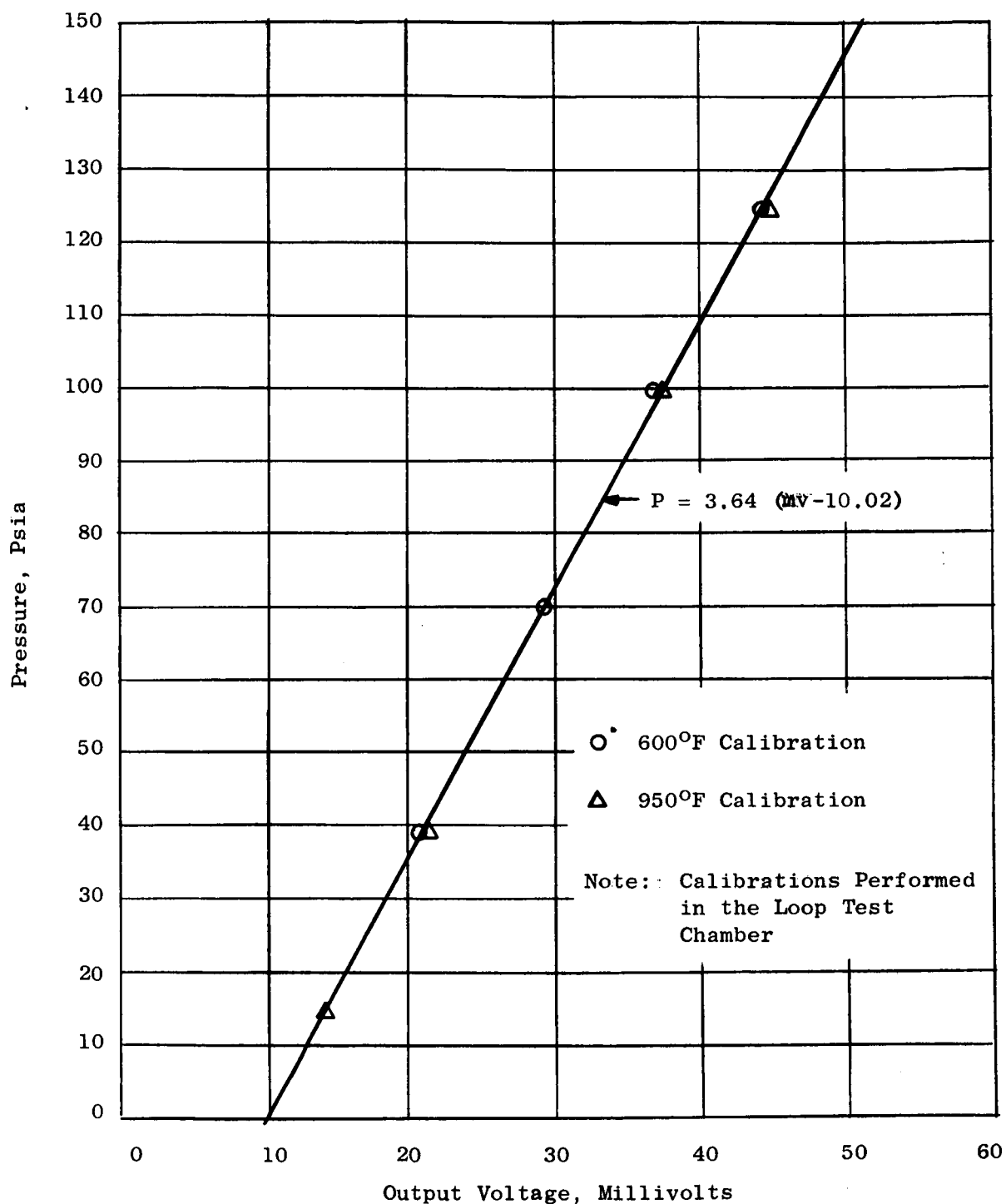
Figure 11. Calibration of the Prototype Corrosion Loop Pressure Transducer That was Used to Measure the Potassium Boiler Outlet Pressure. Calibration was Performed Following Completion of the 5000-Hour Test.
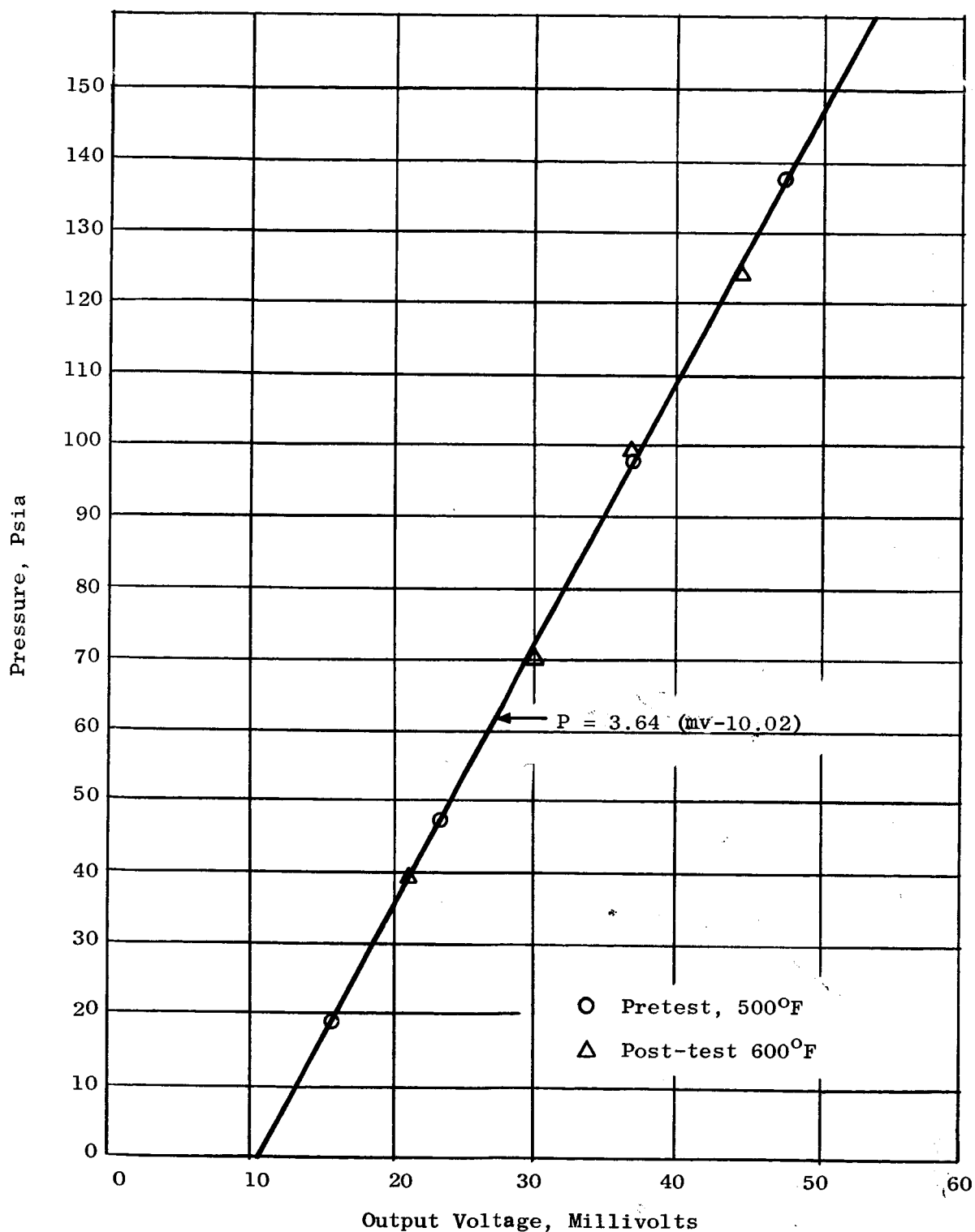
Figure 12. Comparison of the Pretest and Post-test Calibration of the Prototype Corrosion Loop Pressure Transducer Used to Measure Boiler Outlet Pressure.

H. DISASSEMBLY AND EXAMINATION OF LOOP COMPONENTS

The diassembly of the Prototype Corrosion Loop was started immediately after the completion of the distillation and removal of the residual alkali metal from the loop as described in Section F of this report. Following the opening of the vacuum chamber, the thermocouples, valve operating flexible cables and electric power leads were disconnected to permit the transfer of the spool piece to the floor of the test area to facilitate the disassembly of the loop proper into its various components. It was also necessary to remove the EM pump electrical windings (stators) before moving the spool piece. This was accomplished by removing the stator flange bolts and rolling the stators back along the overhead trolleys. The stainless steel sodium pump duct vacuum can is shown in Figure 13 following removal of the windings. Prior to moving the spool piece, the potassium and sodium fill lines and the argon pressurization lines were cut with a cold weld pinch off device which sealed the loop under a positive argon pressure. The pinch off device used was described in an earlier progress report[18].

The procedure used to transfer the loop and spool section to the dis-assembly area was as follows. The bolts connecting the lower spool section flange to the sump flange were removed first. The bell jar was then lowered to the spool section and bolted to the upper spool section flange. The bell jar and spool section (with the loop) were hoisted vertically approximately 10 inches with the overhead hoist to allow a 2-ton fork lift truck to position a cushioned pallet under the spool section. The spool and bell jar were then lowered to the pallet. The hoisting bolts joining the spool to the bell jar were removed and the bell jar alone was hoisted up to clear the top of the loop. The spool and loop were then moved from the test site to the floor of the disassembly area by the fork lift truck.

The first phase of loop disassembly was the removal of all thermocouples and thermal insulation. The thermocouples were removed intact and packaged with their identifying number. Selected specimens of the Cb-1Zr foil insula-tion were saved and packaged for contamination studies. The components in the lower portion of the loop circuits are shown in Figure 14 following removal of insulation and thermocouples.

The second phase of the disassembly was the division of the loop into its major components. The procedure used was to cut the connecting tube near each component with the cold weld pinch off device that sealed the tube as the cut was made. This precaution was taken to allow a more careful exam-ination of the components for residual alkali metal which may not have been removed during the post-test distillation.

The sodium and potassium pump ducts and the insulation assemblies which surrounded them were removed from the stainless steel cans which form the vacuum seal to the chamber wall. The two Cb-1Zr pump ducts are shown in Figure 15 following removal of the 20 layers of Cb-1Zr foil used to insulate the pump ducts from the stainless steel vacuum cans which surrounded them. In order
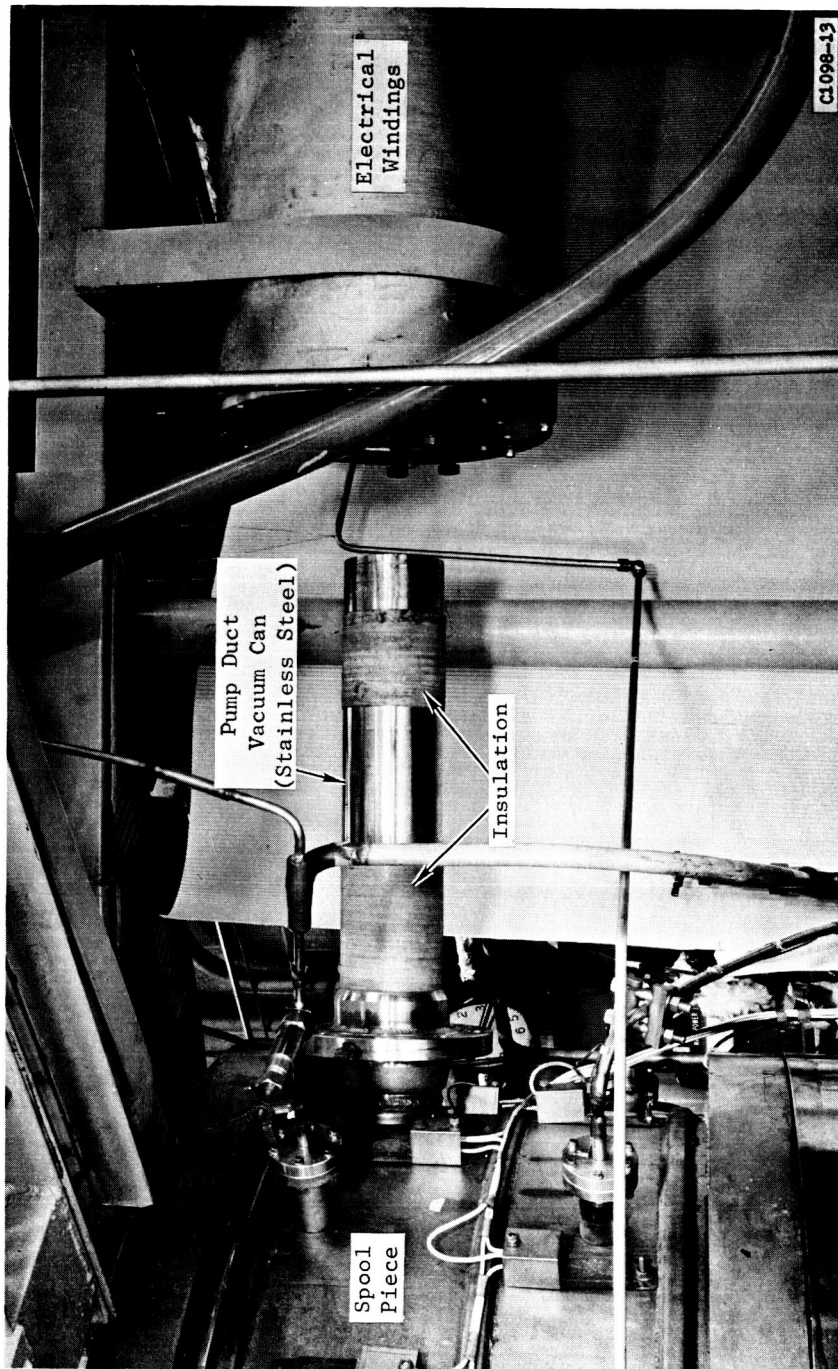
Figure 13. Sodium Pump After Removal of Electrical Windings Following Completion of 5000-Hour Test. (Orig. C6041409)
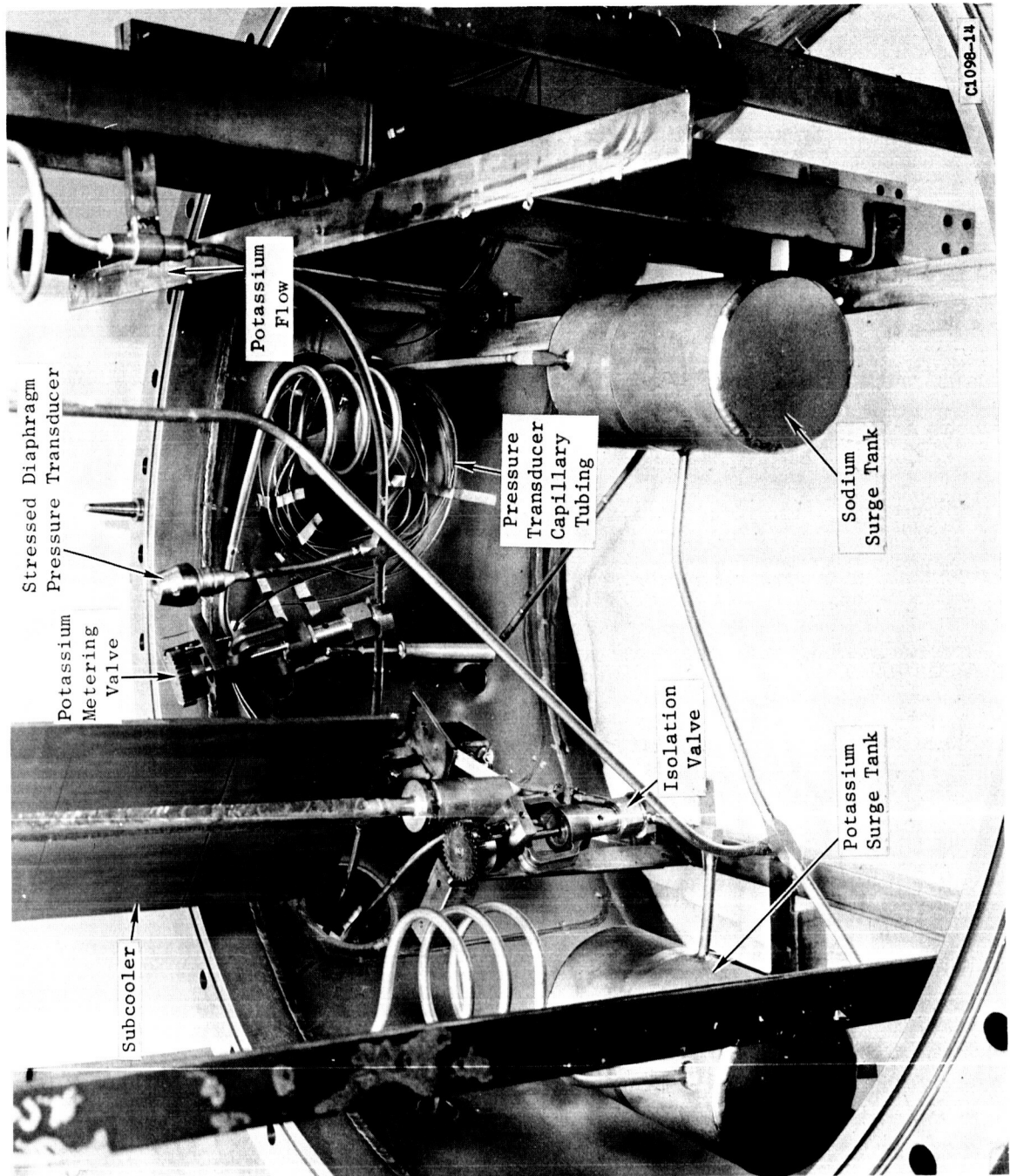
Figure 14. Lower Portion of the Prototype Corrosion Loop Following Removal of Insulation and Condenser Shield Assembly. (Orig. C66050425)
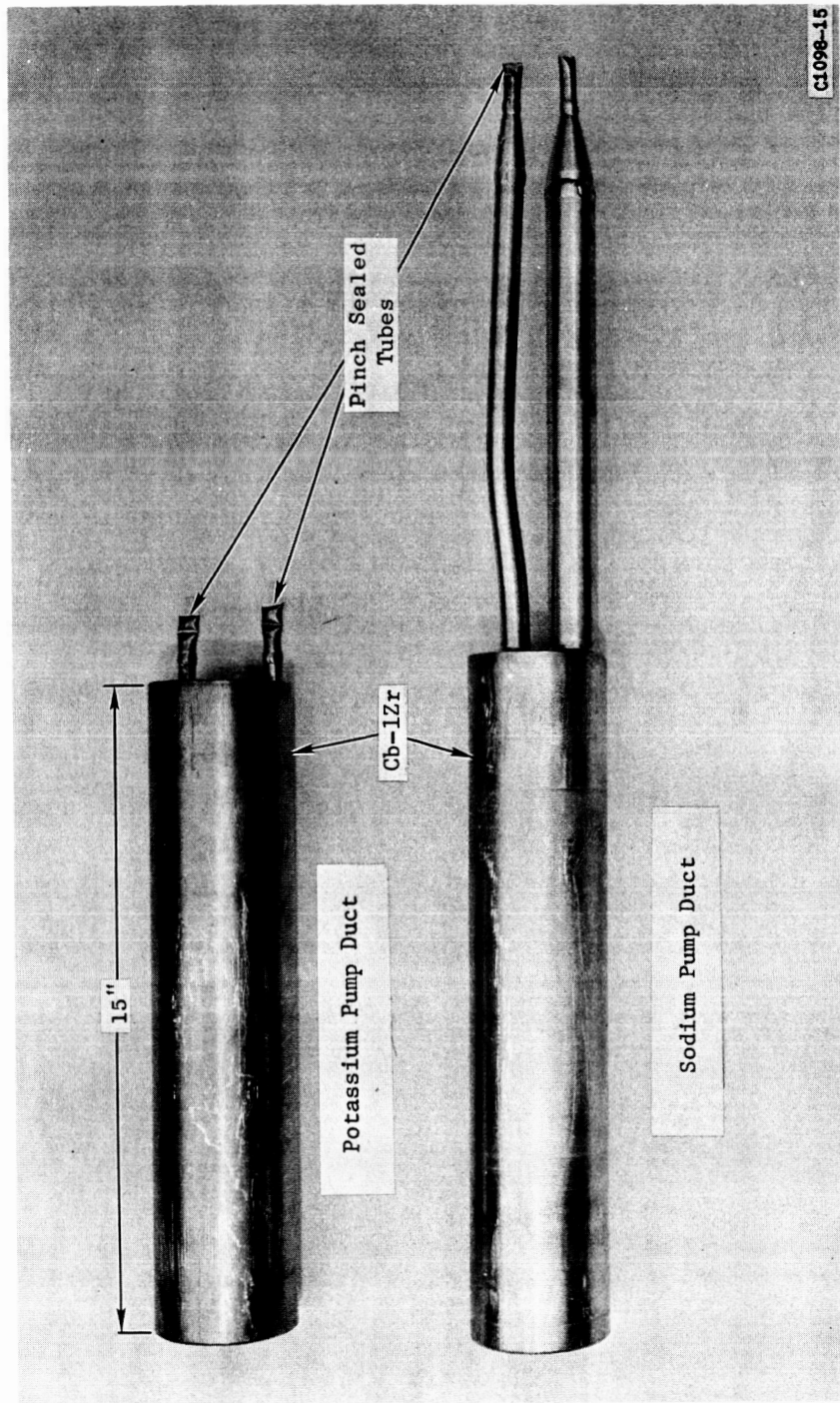
Figure 15. Potassium and Sodium Pump Ducts of the Prototype Corrosion Loop Following Separation From the Loop Circuits and Removal of the Vacuum Enclosure Cans and Cb-1Zr Insulating Foil. (Orig. C66042713)

to determine if any corrosion had occurred in the potassium pump, it was cut open to expose the helical portion for visual examination. This was accomplished by first removing the Cb-1Zr end caps. A small amount of residual potassium was detected after removal of these end caps and this was removed by immersing the pump duct in butyl ethyl "Cellosolve."* One half of the duct can was removed by milling two longitudinal slots to within a few mils of the helical duct and then forcing the slots apart. The pump ducts parts are shown in Figure 16 following disassembly. All parts of the pump appeared to be unaffected by the 5,000-hour test.

Examination of the iron titanate (Fe_2TiO_5) coating on the Cb-1Zr condenser fins indicated that no deterioration or color change had occurred during the 5,000 hours of loop operation. A portion of the condenser and an untested specimen are shown in Figure 17. The temperature of the 8-inch wide by 60-inch long fin varied from 1400°F in the hottest areas closest to the condenser tube to about 800°F in the cooler regions at the outer edges of the fins.

As discussed in Section C of this report, the post-test evaluation of the operation characteristics of the metering and isolation valves indicated some binding in the valve gear drive system. During the disassembly, the valve drive system was carefully examined to determine the cause of this difficulty. The metering valve assembly is shown in Figure 18 following removal from the potassium circuit of the loop.

Visual examination of the stainless steel drive gears of both the isolation and metering valve revealed that galling has occurred between the pinion gear and the spur gear as shown in Figure 18. This galling was responsible for the difficult and erratic valve operation observed on several occasions when valve adjustments were attempted. The cause of the galling was a misalignment of the pinion gear due to an excessive tolerance in the fit of the pinion gear shaft in the tungsten carbide bushing. A design change has been issued to correct this problem and the valve operation problem should be eliminated by this change. The bellows and plug assembly was then removed from the valve body. These components are shown in Figure 19. No evidence of wear or erosion was visible on either the Mo-TZM alloy plug or the Cb-1Zr seat after 5,000 hours at 1100°F and a potassium liquid velocity of 17 ft/sec.

The most critical portion of the Prototype Loop disassembly sequence was the removal of the Mo-TZM alloy nozzle and blade specimens from the Cb-1Zr casing of the turbine simulator. One requirement of the test was to obtain weight change data on the nozzles and blades. This requirement resulted in a design which incorporated a groove along the length of the nine-stage turbine simulator and the Cb-1Zr casing. A Cb-1Zr wire was inserted into this groove during assembly as illustrated in Figure 20. This wire served as a key in preventing rotation of the individual nozzles in the casing during test operation, but its primary purpose was to facilitate disassembly of the turbine simulator without damaging the nozzles and blades. By making a longitudinal cut through the Cb-1Zr casing over the wire, it was possible to spring open the casing and free the Mo-TZM specimens without cutting into them. The milled groove in the casing is shown

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* Trademark.

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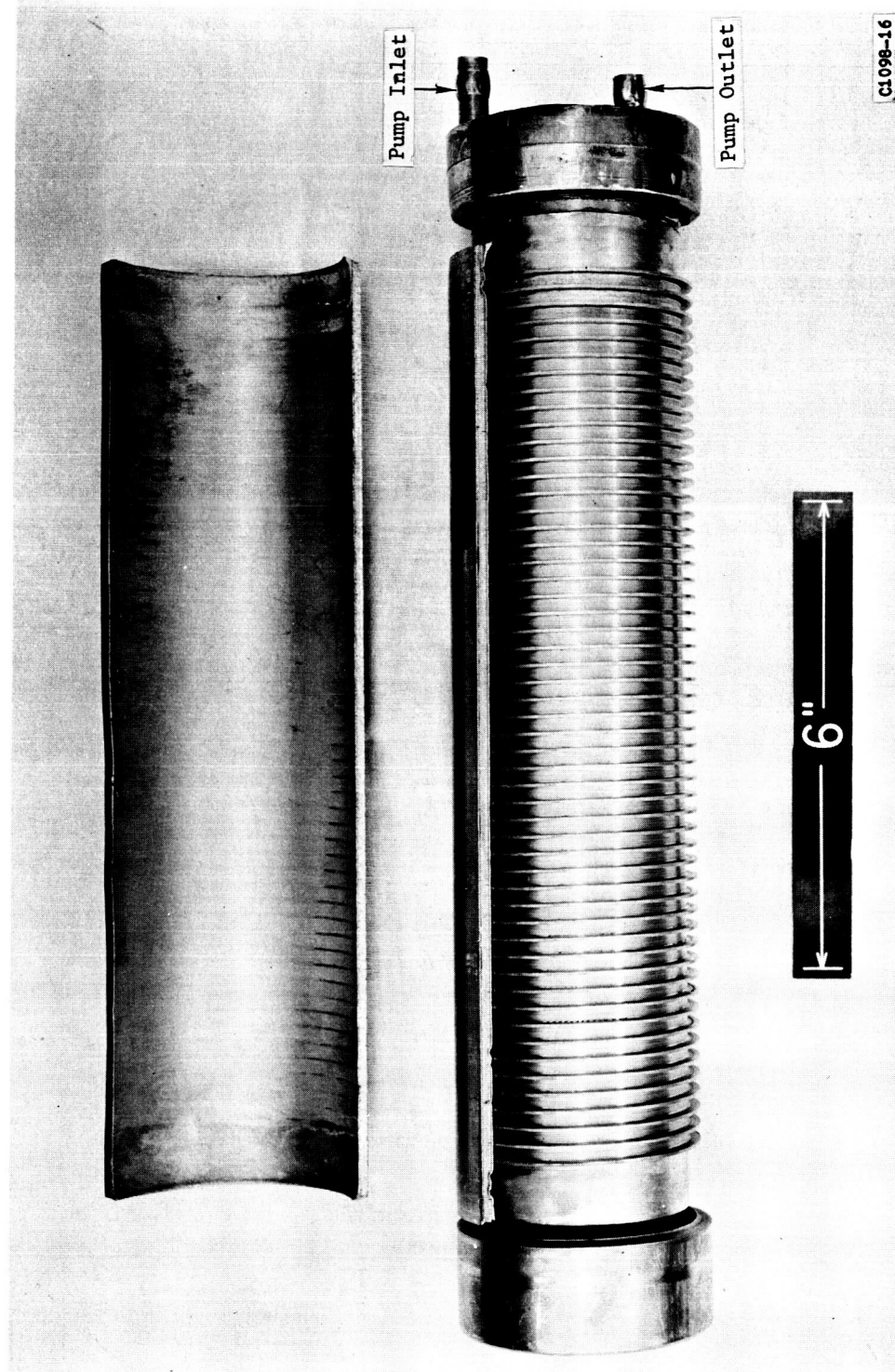
Figure 16. Potassium Pump Duct of the Prototype Corrosion Loop After Removal of Half of the Pump Duct Can Following Completion of the 5000-Hour Test. (Orig. C66052710)
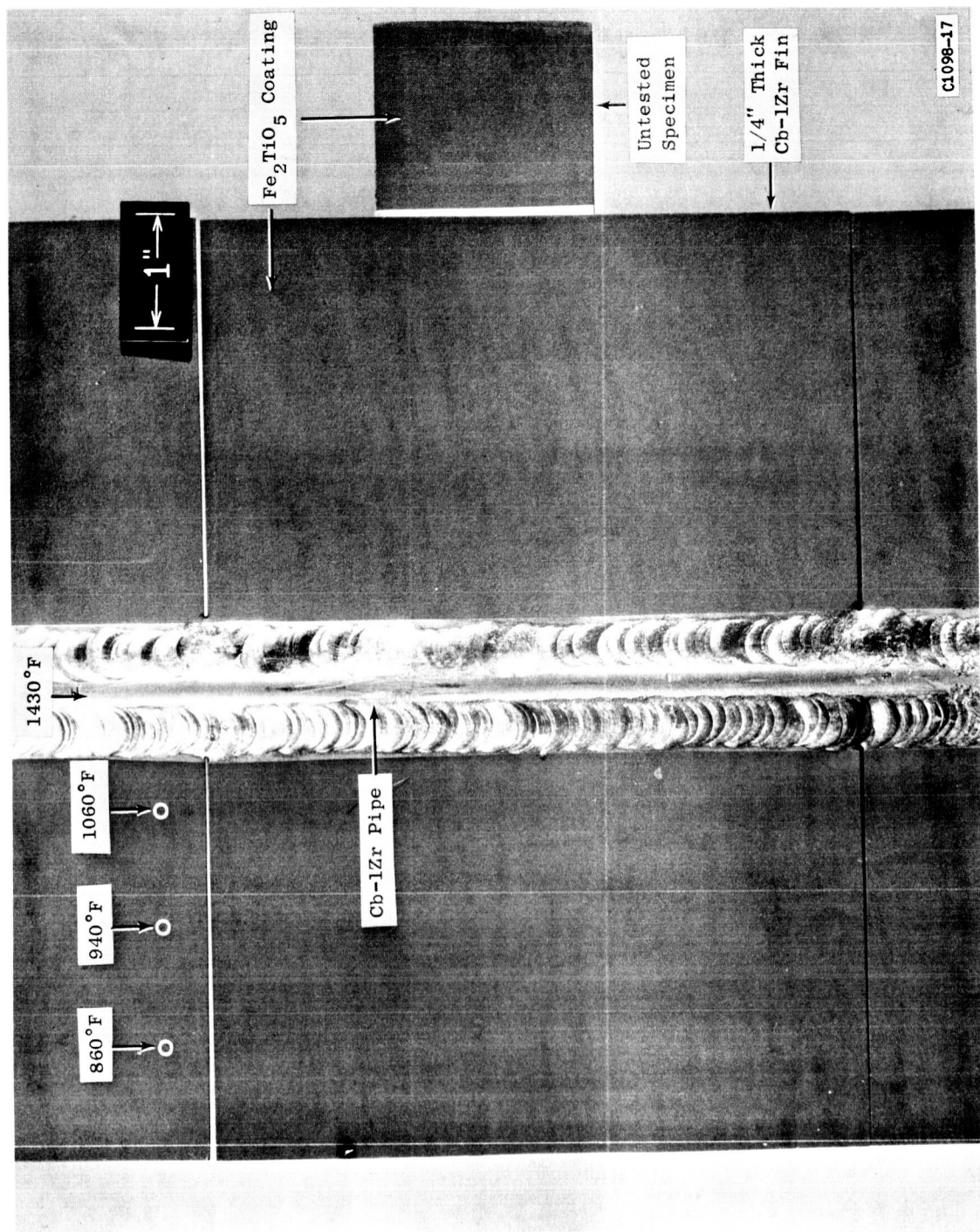
Figure 17. Condenser Pipe and Fin of the Prototype Corrosion Loop Following Completion of 5000-Hour Test. (C66050428)
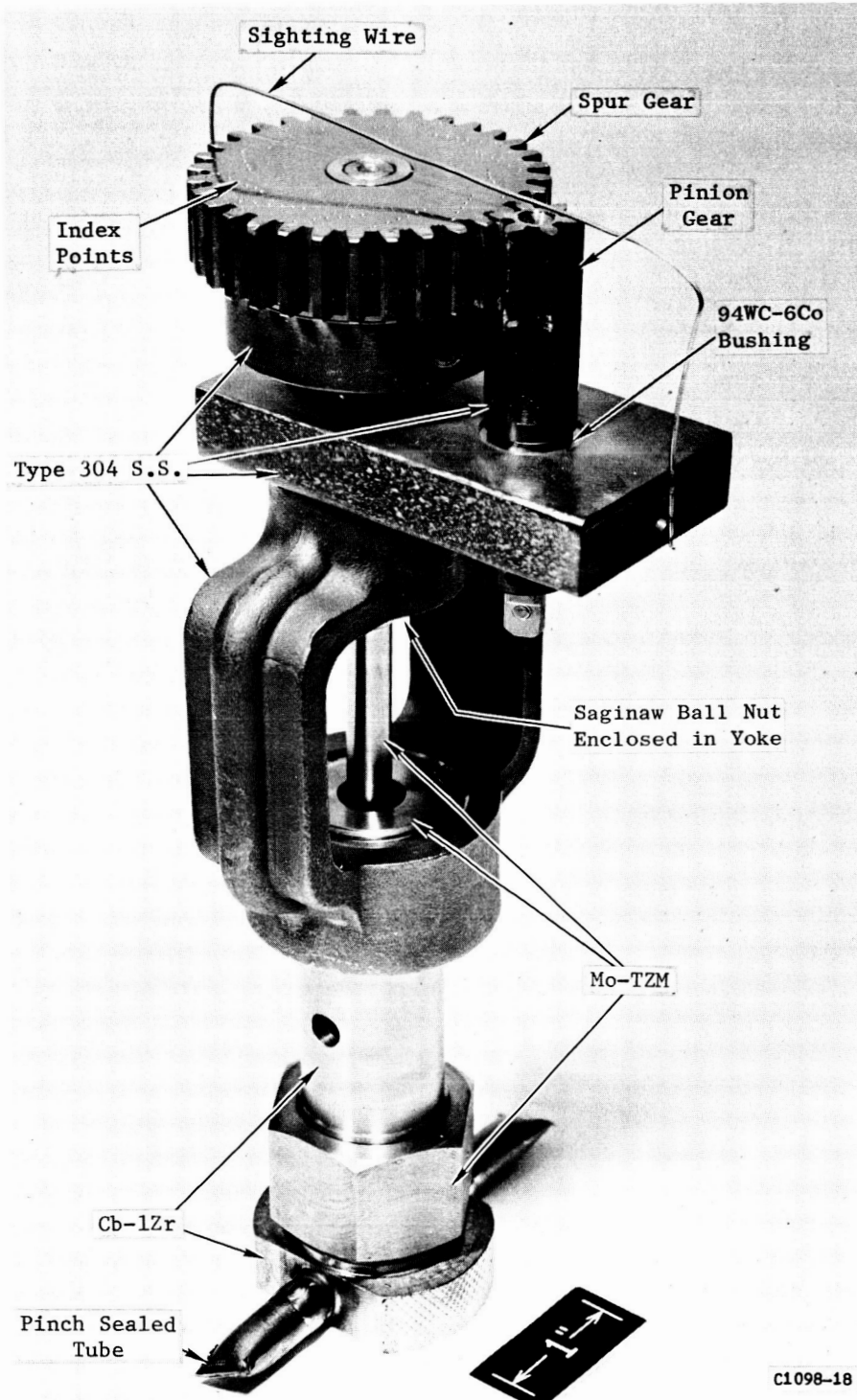
Figure 18. Metering Valve Following Completion of the 5000-Hour Test.
(Orig. C66042917)
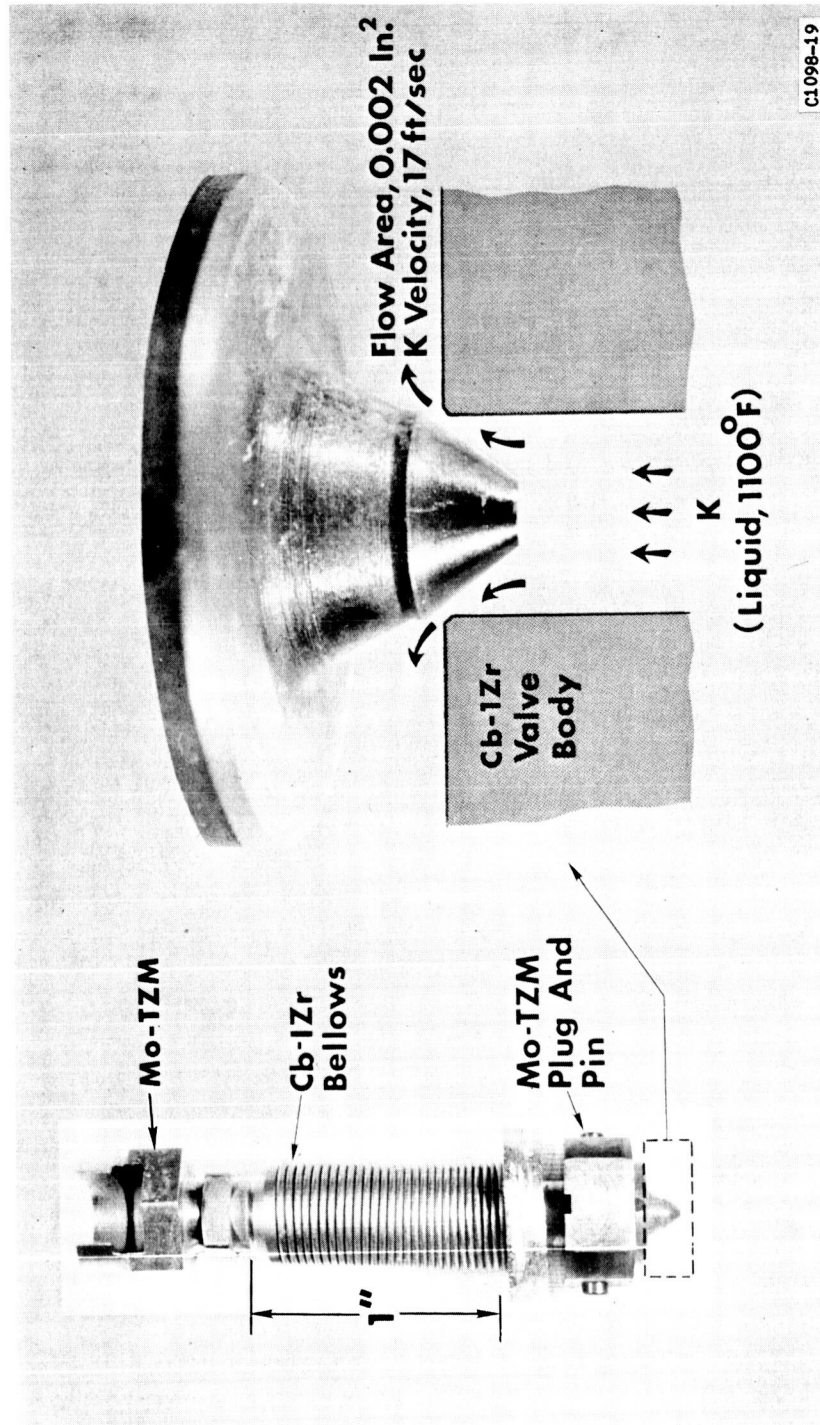
Figure 19. Bellows and Plug Assembly of the Prototype Corrosion Loop Metering Valve Following 5000-Hour Test. (Orig. C6605194B, C6605194B)

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Labels on figure:
- K
- Support Bracket
- Thermocouple Well
- Mo-TZM Nozzles and Blades
- To Pressure Transducer
- 6"
- Alignment Wire
- To Pressure Transducer
- K to Condenser
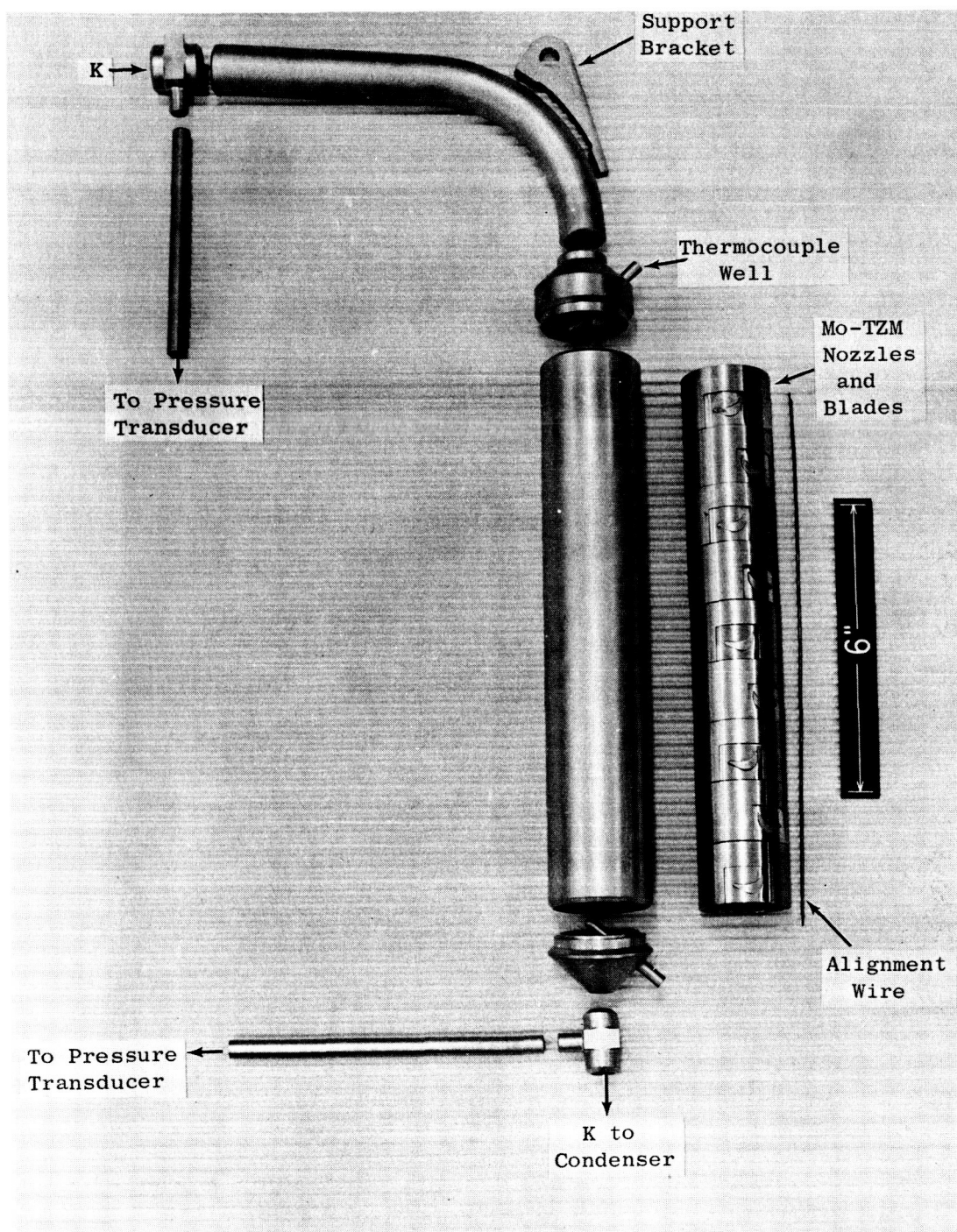
C1098-20

Figure 20. Turbine Simulator (Stages 2-10) of the Prototype Corrosion Loop Prior to Assembly. Nozzles and Blades: Mo-TZM. Tubing, Reducers, Casing and Miscellaneous Fittings: Cb-1Zr.

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-45-

in Figure 21. The enlarged view in this illustration shows the Cb-1Zr wire and the separation of the casing. Removal of the stack of nine nozzles and blades from the casing was done by spreading the slot approximately 1/16-inch and gently tapping the casing. The specimens slipped from the casing with no evidence of bonding between the casing and the specimens. A description of the specimens and the results of the weight change determinations are given in the next section of this report.

I. PRELIMINARY EVALUATION OF TURBINE SIMULATOR COMPONENTS

Following the removal of the Mo-TZM alloy turbine simulator nozzles and blade specimens from the Cb-1Zr containment casings, as described above, visual examinations at a magnification of 30 diameters were performed. The entrance side of the ten nozzles and the blade-blade pad assemblies are shown in Figure 22. The exit sides of the nozzles are shown in Figure 23. With the exception of a few small slightly darkened areas on the exit side of several of the blades, the specimens looked just as they did before the test. No evidence of impingement damage could be detected on any of the ten blade specimens. It may be noted in Figure 22 that the only blade specimen which gave a clear indication of the impingement area was the blade in Stage No. 1. Enlarged views of the Stage No. 1 components are given in Figure 24 to illustrate the excellent condition of the components from the stage which showed the most discoloration of the impingement area. The results of weight change measurements on the nozzles and blades are given in Table VI and indicate that no significant weight changes occurred.

J. CHEMICAL ANALYSIS RESULTS ON LOOP COMPONENTS

Results have been received on several specimens of loop components and these preliminary results are reported below.

Very thin refractory metal specimens exposed in high temperature, high vacuum environments are very sensitive indicators of the extent of contamination by elements such as oxygen, nitrogen, hydrogen and carbon. The 0.002-inch thick Cb-1Zr foil used to insulate the loop was ideal for this purpose. Samples of foil from the single layer of insulation that surrounded the nine-stage turbine simulator were submitted for analysis. The results obtained on these specimens are given in Table VII. It may be noted that only the oxygen concentration of the foil changed significantly during the 5,000-hour test. Although the increase of 546 ppm oxygen is significant, it should be noted that this amount of pickup would amount to less than 20 ppm in a 0.060-inch thick tube wall, even if all the contamination is assumed to occur from one side and less than 10 ppm if two-side contamination of the foil is assumed.

Results have also been obtained on specimens of the 1-inch OD x 0.1-inch wall Cb-1Zr pipe from the potassium vapor carryover line located between the single-stage and the nine-stage turbine simulators.

The tube samples were taken from adjacent regions that operated at 1780°F during the 5,000-hour test. One of the locations was wrapped with Cb-1Zr foil insulation and the other was uninsulated. The results obtained are listed in Table VIII and are indicative of the very low levels of contamination achieved in this experiment. The averages of the dupicate analyses performed
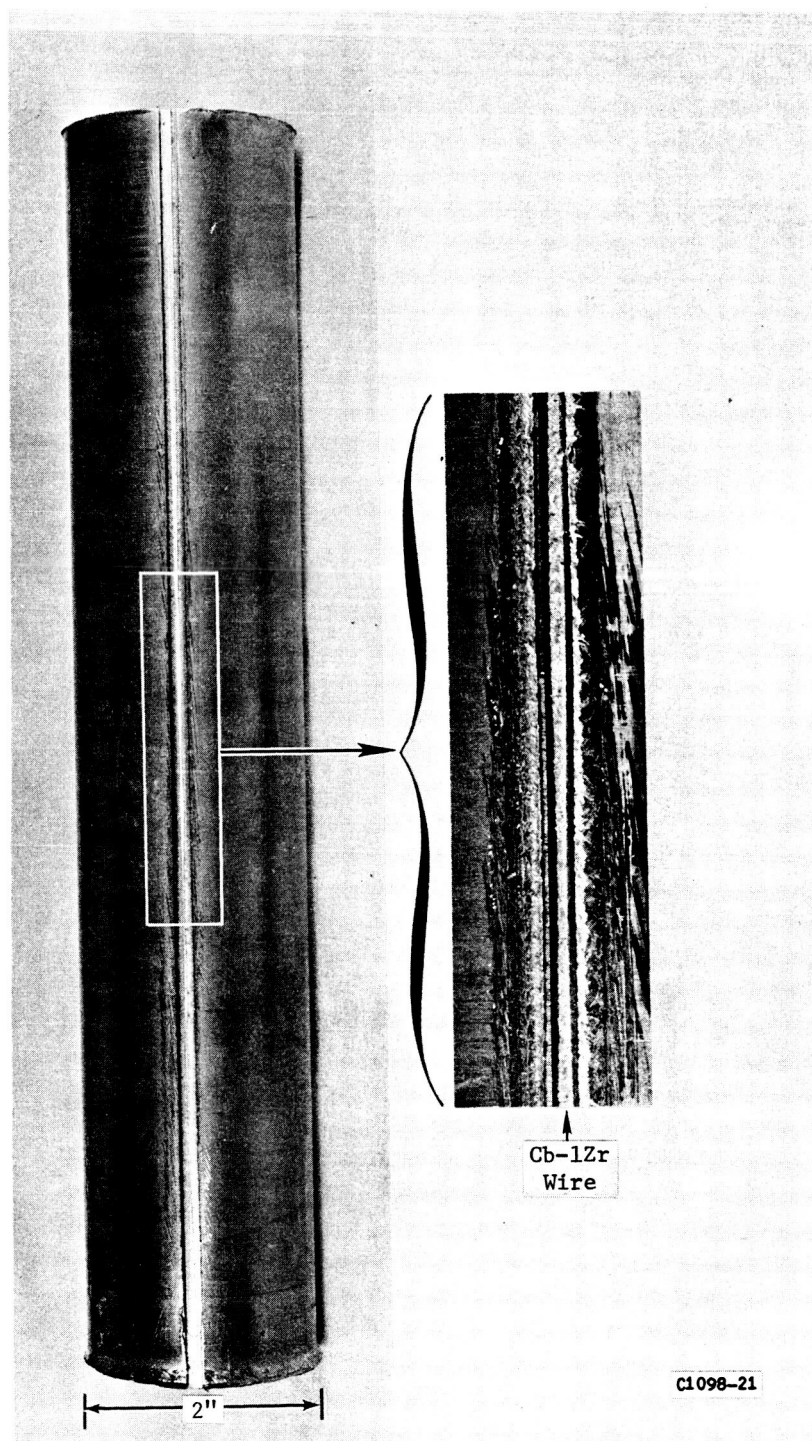
Figure 21. Cb-1Zr Casing of Nine-Stage Mo-TZM Alloy Turbine Simulator Showing Milled Groove and Cb-1Zr Alignment Wire Just Prior to Removal of Simulator Nozzles and Blades. (Orig. C66050517, C66050526)
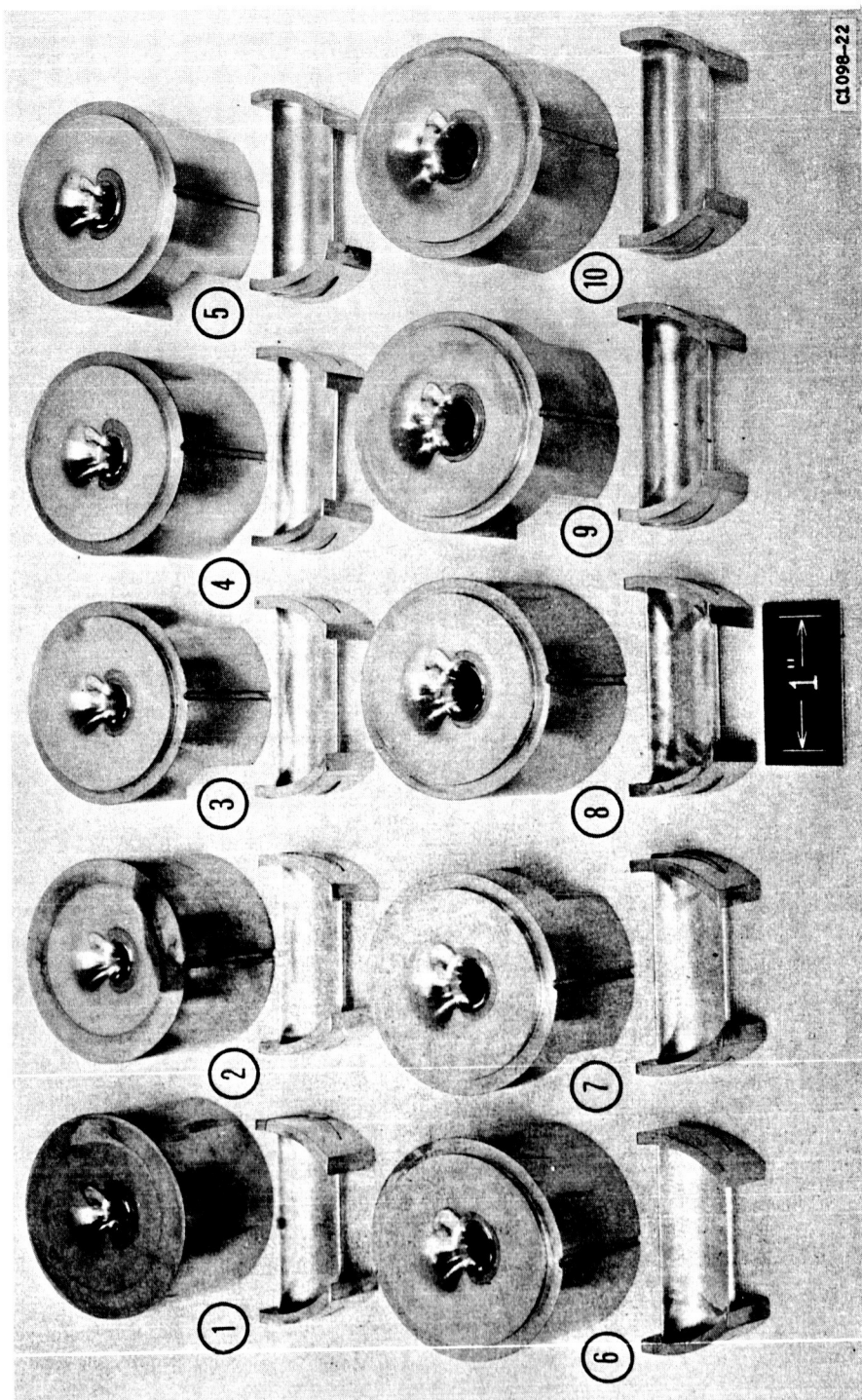
Figure 22. Mo-TZM Alloy Turbine Simulator Nozzles, Blade Specimens and Specimen Holding Pads From the Prototype Corrosion Loop Following 5000-Hour Exposure to Potassium. (Orig. C66050510)
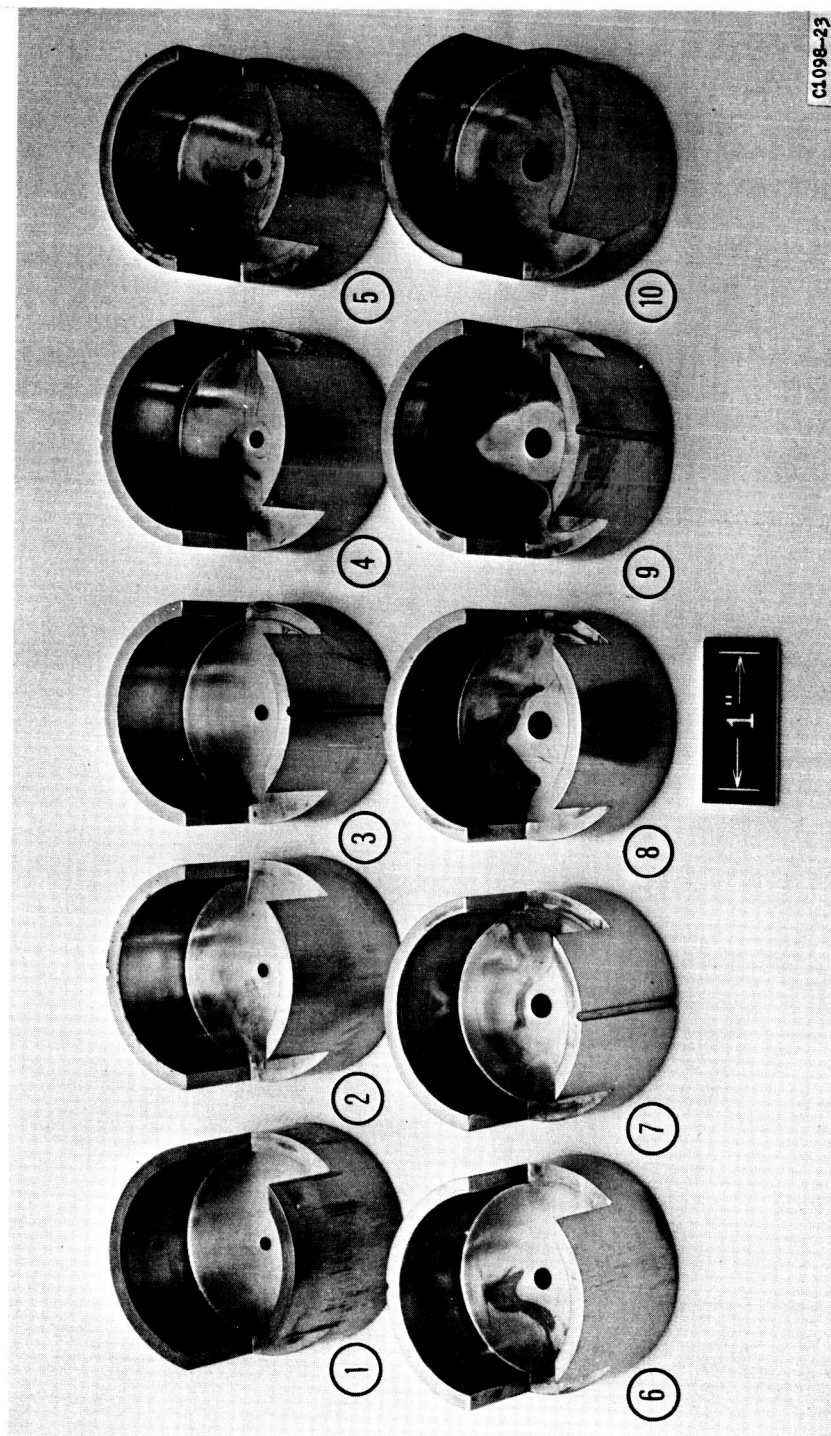
Figure 23. Outlet Side of Nozzles of Turbine Simulator Stages No. 1 thru No. 10. Nozzle Throat Diameters Increase From Nozzle No. 1 (Upper Left) to No. 10 (Lower Right). (Orig. C66050515)

Convex Side of Blade

K →

1.75"

Concave Side of Blade

K →

0.1"

Concave Side of Blade
Showing Impingement Edge

Concave Side of Blade

K →

Exit Side of Nozzle

Entrance Side of Nozzle

1.75"

C1098-24
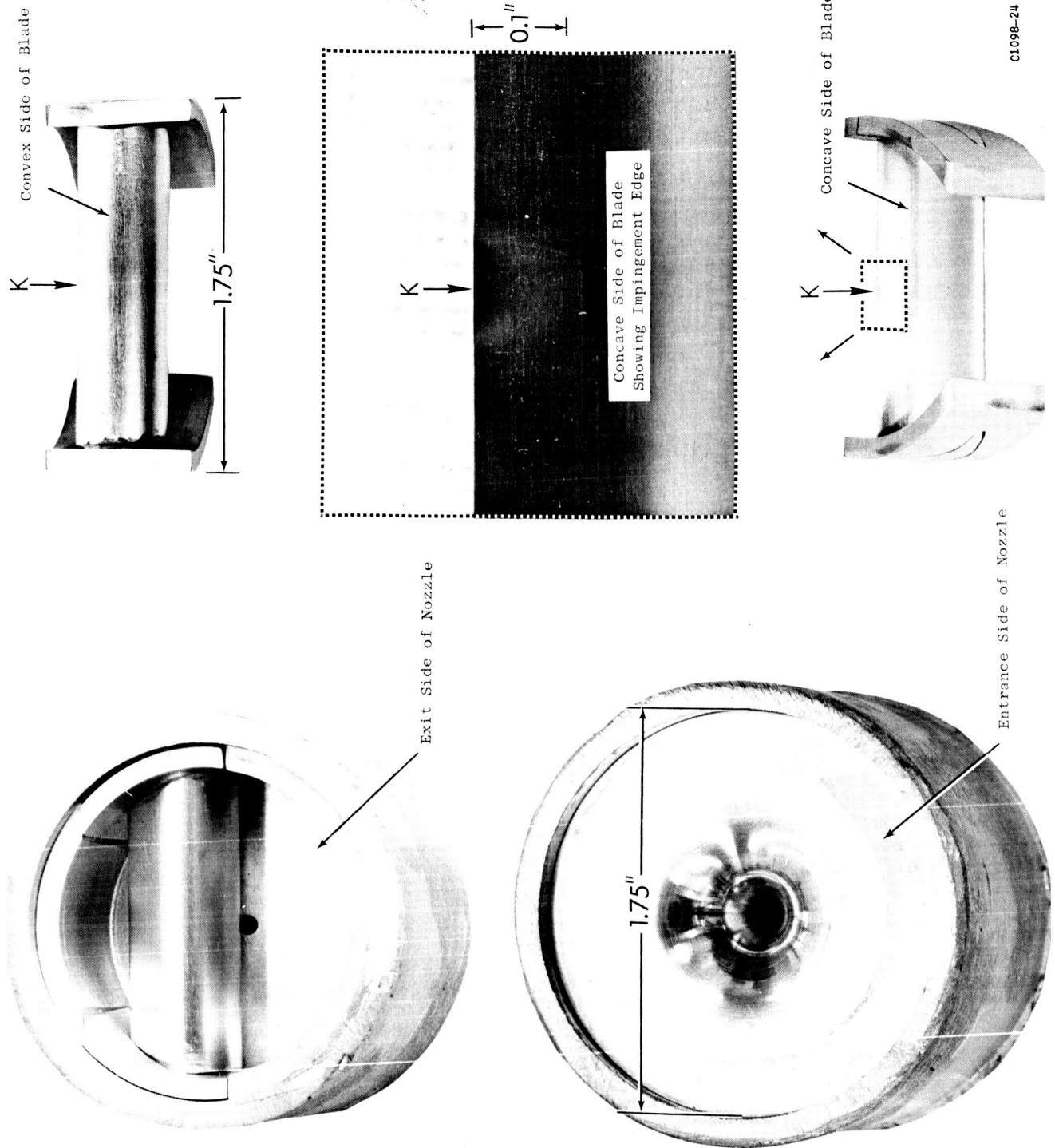
Figure 24. Turbine Simulator Stage No. 1 From the Prototype Corrosion Loop
Following the 5000-Hour Test.

TABLE VI.   SUMMARY OF PROTOTYPE CORROSION LOOP TURBINE SIMULATOR TEST
CONDITIONS AND RESULTS OF WEIGHT CHANGE DATA OBTAINED ON Mo-TZM ALLOY
NOZZLES AND BLADE SPECIMENS FOLLOWING COMPLETION OF 5,000-HOUR TEST

Stage No.	Nozzle Inlet Temperature, °F	Impingement Velocity, ft/sec	Quality	Weight Change[1], g	
				Blade	Nozzle
1	2000	1125	138° superheat	-0.002	-0.001
2	1785	1350	88%	0	+0.008
3	1740	1200		+0.001	+0.006
4	1700	1110		+0.001	+0.008
5	1660	1080		+0.001	+0.009
6	1620	1015		0	+0.006
7	1575	1045		0	+0.008
8	1535	845		-0.001	+0.007
9	1490	860		0	+0.008
10	1450	830		0	+0.007

[1] Nominal weight of blade specimens was 25 g; nominal weight of nozzles was
255 g. Weight change data obtained to four decimal places and reported
to nearest mg.

TABLE VII. RESULTS OF CHEMICAL ANALYSIS OF Cb-1Zr FOIL
TAKEN FROM THE SINGLE LAYER OF INSULATION THAT
SURROUNDED THE MINE-STAGE TURBINE SIMULATOR
DURING THE 5,000-HOUR PROTOTYPE
CORROSION LOOP TEST

Specimen	Chemical Analysis, ppm(1)			
	O	N	H	C
Before Test	244	50	3	85
Top Region (1400°F)	790	61	6	110
Bottom Region (1130°F)	750	45	12	120

(1) Results given are the average of duplicate analyses performed
     on the 0.002-inch thick foil.

TABLE VIII. RESULTS OF CHEMICAL ANALYSIS OF Cb-1Zr
TUBING[1] FROM THE VAPOR CARRYOVER LINE OF THE
PROTOTYPE CORROSION LOOP FOLLOWING
THE 5,000-HOUR TEST

Specimen	Chemical Analysis, ppm			
	O	N	H	C
Before Test	245	46	10	40
Uninsulated Tube[2]	300	51	5	46
Insulated Tube[3]	304	56	5	30

(1) One-inch OD x 0.1-inch wall; tube temperature during test
was 1780°F.

(2) Average of two analyses.

(3) Average of four analyses.

show increases of approximately 80 ppm oxygen and 70 ppm carbon in the uninsulated region with no changes in either nitrogen or hydrogen concentrations. It is interesting to note that the uninsulated tube, which did have the benefit of the shielding effect of the Cb-1Zr foil, showed no significant contamination. The low increases in oxygen and carbon concentrations are consistent with the results obtained on specimens of the 0.002-inch thick Cb-1Zr foil reported above.

IV. FUTURE PLANS

A. Evaluation of the Prototype Corrosion Loop Components will be continued.

B. Preparation of topical reports covering the various portions of the program which have been completed will continue.

APPENDIX A

DRAINING, SAMPLING AND FILTERING OF THE POTASSIUM

AND SODIUM CIRCUITS OF THE PROTOTYPE CORROSION LOOP

The post-test procedures used to prepare the transfer and sampling systems prior to the transfer of the alkali metals from the loop surge tanks after distillation of the alkali metals from the loops, and the subsequent sampling and filtering of the alkali metals are described in detail in the following section.

PROTOYPE SYSTEM

POTASSIUM LOOP SAMPLING PROCEDURE

Operation and Purpose

Prior to draining the potassium into the transfer system charge pot, the following operations were carried out:

1. Attached high vacuum sampler and filtration system to the transfer system to obtain samples and filter most of the alkali metal into the disposal tank.

    (a) Cleaned, assembled and attached high high vacuum sampler. See schematic diagram (Figure 25).

    (b) Assembled 5-micron filter by TIG welding with backup gas.

    (c) Installed the filter and transfer line between valves U and W with swaglok and conoseal connections where possible—otherwise welded with backup gas.

    (d) Helium leak checked all connections of the assembly. Maximum allowable leakage = 5×10^{-10} std cc/sec.

2. Blew residual alkali metal from transfer lines into the disposal tank to minimize contamination of the test metal with the fill metal.

    (a) Evacuated the transfer system lines through valve Z with valves FF, W, S, DD and the bottom sample tube valve closed (sampler and transfer line were already under vacuum from leak checking operation).

    (b) Heated all transfer system lines to $250 \pm 30°F$ and outgassed the sampler until the pressure rise rate was less than one micron-liter per minute.

    (c) Closed valves X and GG and pressurized the sampler and charge pot to 15 psia. See Figure 25.

Figure 25. Schematic Diagram of the Prototype Corrosion Loop Drawing, Sampling and Filtering System.

Operation and Procedure

Procedure

(d) Opened valve GG and blew argon through valves GG, T and V into the disposal tank until pressures equalized.

(e) Opened valve X, closed valve T and again evacuated the disposal tank.

(f) Closed valve X, pressurized the sampler to 15 psia and blew argon through the valve on the bottom of the sample tube through valves U and V into the disposal tank until the pressures equalized.

(g) Opened valve X, closed sample tube bottom valve and evacuated transfer system lines through valve Z.

3. Drained potassium into the transfer system charge pot as previously indicated.

4. Obtain potassium sample from charge tank.

4.

(a) Closed valves FF, DD, GG, V, W, S and the valve on the bottom of the sample tube. See Figure 25. Double checked that valves V and W were closed.

(b) Closed valves Y and X with the sampler and disposal tank evacuated.

(c) Opened valve GG, opened the valve on the bottom of the sample tube and flushed about 100 cc of potassium into the sampler reservoir.

(d) Closed the valve on the bottom of the sampler tube, opened valve Y to pressurize the sampler and cooled the sample to room temperature.

5. Filtered the remaining potassium into the disposal tank.

   5. (a) Pressurized the charge pot to 15-17 psia.

      (b) Opened valve W and filtered the potassium in the charge pot into the disposal tank. Filtration was complete when a rapid pressure drop occurred, followed by a constant pressure reading.

      (c) Closed valve W, opened valve X and cooled transfer system to room temperature at 15-17 psia after sampling and filtering potassium charge.

PROTOTYPE SYSTEM

SODIUM LOOP SAMPLING PROCEDURE

Operation and Purpose

Prior to draining the sodium into the transfer system charge pot, the following operations were carried out.

1. Attached high vacuum sampler and filtration system to the transfer system to obtain samples and filtered most of the alkali metal into the disposal tank.

2. Flow residual alkali metal in transfer lines into the disposal tank to minimize contamination of the hot metal with the fill metal

Procedure

1. (a) Cleaned, assembled and attached high vacuum sampler. See Figure 25.

   (b) Assembled 5-micron filter by TIG welding with backup gas.

   (c) Installed the filter and transfer line between valves U and W with swagelok and conoseal connections where possible – otherwise welded with backup gas.

   (d) Helium leak checked all connections of the assembly. Maximum allowable leakage = 5 x 10^{-10} std cc/sec.

2. (a) Evacuated the transfer system lines through valve Z with valves FF, W, S, DC and the bottom sample tube valve closed (sampler and transfer line were already under vacuum from leak checking operation).

   (b) Heated all transfer system lines to 250 $\pm$ 30°F and outgassed the sampler until the pressure rise rate was less than one micron-liter per minute.

   (c) Closed valves X and GG and pressurized the sampler and charge pot to 15 psia.

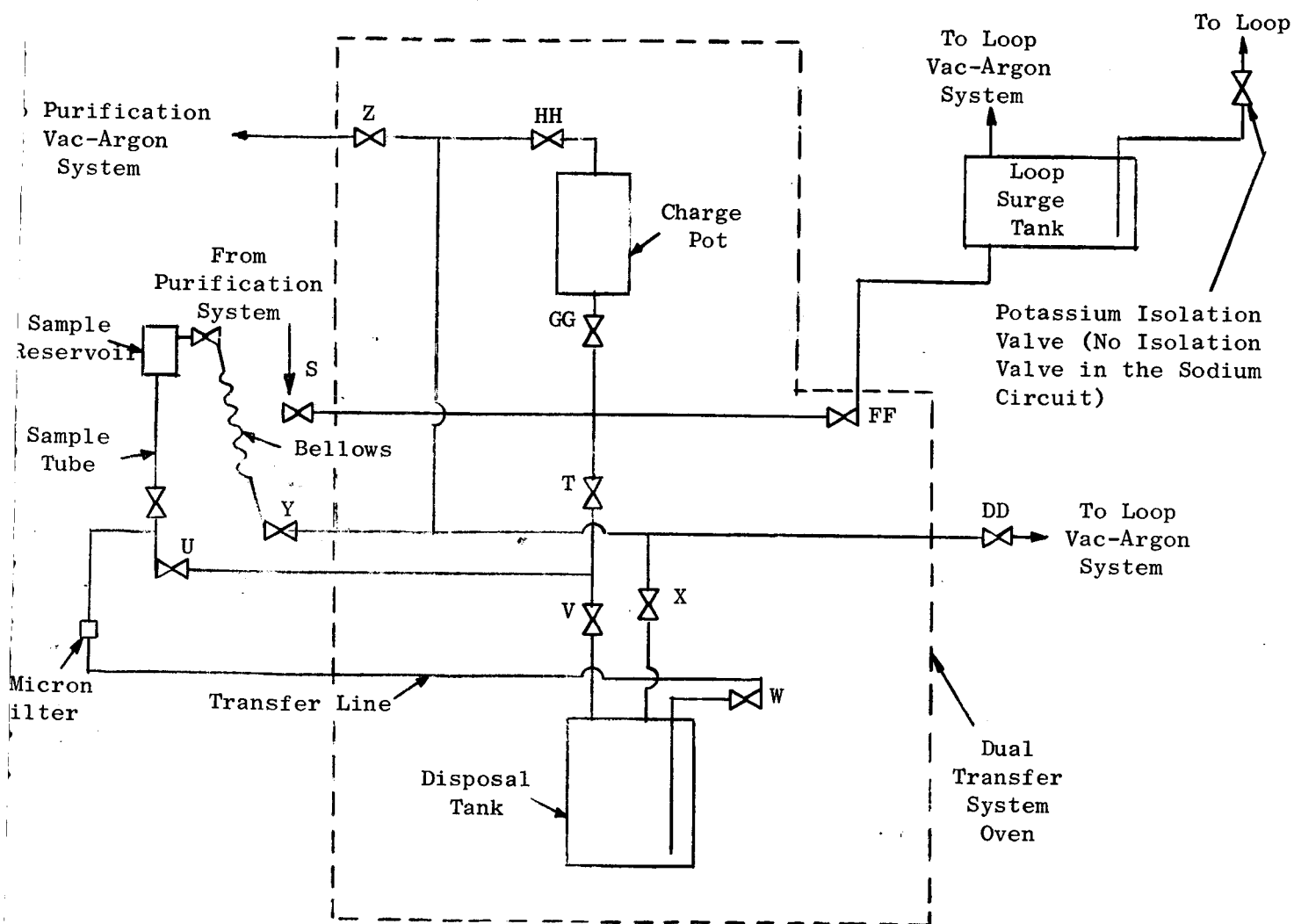## Operation and Purpose

3. Drained sodium into the transfer system charge pot as previously indicated.

4. Obtained sodium sample from charge tank.

Procedure

(d) Opened valve GG and blew argon through valves GG, T and V into the disposal tank until pressures equalized.

(e) Opened valve X, closed valve T and again evacuated the disposal tank.

(f) Closed valve X, pressurized the sampler to 15 psia and blew argon through the valve on the bottom of the sample tube through valves U and V into the disposal tank until pressures equalized.

(g) Opened valve X, closed sample tube bottom valve and evacuated transfer system lines through valve Z.

4. (a) Closed valves FF, DD, GG, V, W, S and the valve on the bottom of the sample tube. See Figure 25. Double checked that valves V and W were closed.

(b) Closed valves Y and X with the sampler and disposal tank evacuated.

(c) Opened valve GG, opened the valve on the bottom of the sample tube and flushed about 100 cc of sodium into the sampler reservoir.

(d) Closed the valve on the bottom of the sample tube, opened valve Y to pressurize the sampler and cooled the sample to room temperature.

PROTOTYPE SYSTEM

SODIUM LOOP SAMPLING PROCEDURE

(Cont'd)

Operation and Purpose

5. Filtered the remaining sodium into the disposal tank.

Procedure

5. (a) Pressurized the charge pot to 15-17 psia.

(b) Opened valve W and filtered the sodium in the charge pot into the disposal tank. Filtration was complete when a rapid pressure drop occurred, followed by a constant pressure reading.

(c) Closed valve W, opened valve X and cooled transfer system to room temperature at 15-17 psia after sampling and filtering potassium charge.

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